GRACE

Construction Products Division

TO: H. C. Duecker

DATE: December 9, 1976

FROM: Julie C. Yang (2)

SUBJECT: Asbestos Fiber Counting in the Cambridge Laboratory

cc: H. A. Brown
H. A. Eschenbach
R. H. Locke
F. G. Serafin
J. W. Wolter
R. L. Oliverio/Libby
R. Geiger/Libby
J. E. Foley
J. P. Wallace
File: 71-044

SUMMARY

The reproducibility of standard fiber counts in the Cambridge laboratory was determined. Variables such as operators, equipment, and technologies were included.

It is concluded that the Cambridge laboratory showed a variation of less than 15% of the total fiber counted. The reproducibility in such a range is considered excellent compared with those described in publications (generally 40-50%).

Recent Libby lab samples were also counted in our lab and used in the study for discussion.

BACKGROUND INFORMATION

The counting has been carried out in the Cambridge laboratory by two operators, trained originally by F. G. Serafin. Facilities available for counting is a phase-contrast microscope of Bausch & Lomb, DynaZoon model; and also a TV viewing screen attachment by Techni-Quip Corp., so that an operator can either count the fibers directly with the microscope or count the fibers on the projected TV screen.

It is necessary to know the reproducibility and accuracy of our measurements in order to meet the OSHA and MESA requirements. Unfortunately, as far as we know, there is no primary standard available on the market which will allow us to check the absolute accuracy of our method. The only way we can determine whether we have reliable results is the verification of counting specific samples by several experienced personnel.

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CRITERIA

It is very difficult to decide (1) whether the fiber being counted is a true fiber even though the aspect ratio is greater than 3 to 1, (2) whether the size of fiber should be counted at all. For example, slivers of vermiculite or plates standing on edge should be avoided; the judgment is mainly based on experience and knowledge of microscopy.

Based on Field Information Memorandum #74-92 of OSHA (issued 11/21/74), the maximum diameter of a fiber to be counted is 3 μ, and the maximum length of a fiber to be counted is 30 μ. The Memorandum from MESA issued 12/13/74 is about the same except the maximum length of fiber to be counted is 25 μ.

In the Cambridge laboratory we have used the following guidelines:

1) particles must appear to be fiberous rather than as crystals or slivers,
2) the maximum diameter of a fiber to be counted is 3 microns,
3) the minimum length of a fiber to be counted is 5 microns,
4) the maximum length of a fiber to be counted is 30 microns,
5) the length to width ratio must be 3 or more to 1.
6) the separate or individual fibers must contain fibrils; a fibril cannot be subdivided and would be counted as one if it meets the other criteria.
7) The basic number of fields to be counted is 50, and if no fibers or only one fiber is found in counting the first ten fields, then 100 fields should be counted.

EXPERIMENTAL DATA

All the counting data are presented in Tables 1 to 6. In these tables the average variations in % are calculated and presented.

Table 1. Effect of different viewing equipment with the same operator.
Table 2. Effect of same viewing equipment with different operators:
(a) Microscope (b) TV screen
Table 3. Effect of the same viewing equipment and the same operator.
Table 4. Summarized results of Tables 1, 2, and 3.
Table 5. Statistical study of Cambridge results on counting samples from the Libby lab.
Table 6. Comparison of the results from Libby laboratory and Cambridge laboratory.

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DISCUSSION

Regardless of the variables employed, the Cambridge laboratory showed less than 15% variability in the counting data, and the average standard deviation of 20 samples is 0.18 fiber per cc.

According to a few papers published in this area (Ref. 1 and 2), the standard deviation of the results varied between 0.4 - 1.2 f/cc, under the field conditions, and 0.2 f/cc under ideal laboratory conditions. Another paper cited the coefficient of variation to be about ± 20%, and the maximum can be ± 50%. Based on these results, the Cambridge data looked very respectable.

In verifying the Libby data as shown in Table 6, the Libby counting results are consistently higher (in fact, about 2X) than the Cambridge results. (H. Eschenbach and F. Serafin counted 5 samples and their results are in-between, but closer to the lower values of the Cambridge lab.)

It is possible that the two laboratories are using different criteria to identify the fiber or select the fibers for counting. The more likely explanation of the difference is because the filter Cambridge received were the ones Libby had cut a section off of, for evaluation. In this operation, the filter surface has been disturbed and some fibers may have fallen off resulting in lower fiber values. However, another factor is that in this group of samples (from Libby lab) the range of fiber length was very large, wider than usual; there were many fibers much greater than 30 μ and also many less than 5 μ, but close to 5 μ size. In our procedure these should not be counted.

RECOMMENDATION

To check the discrepancies between Libby and Cambridge laboratories, the following actions are recommended:

1) The Cambridge-prepared slides of Libby samples from T&A 49930 will be sent back to Libby for counting.
2) A second set of Cambridge-prepared slides (T&A 49561-2 samples from Portland, Oregon) will be sent also to Libby for counting. This group has very different fiber length distributions and fiber density than the group from Libby.
3) Libby-laboratory-prepared slides of T&A 49930 will be sent to Cambridge for counting.

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4) The Libby samples (in casettes) for T&A 49930 will be sent back to Libby for re-evaluation, which would show the effect of transportation and filter handling on counting.

5) After all the counting results are compiled, decisions will be made on how to equalize our results.

6) In addition, the calibration factors for both laboratories will be rechecked.

REFERENCES

1. Ortiz, L.W.; Ettinger, H.J.; and Fairchild, C.I., "Calibration Standards for Counting Asbestos" J. Am. Ind. Hygiene Assoc. pp. 104-111 (Feb. 1975)

2. Rajhans, G.S.; and Bragg, G.M. "A Statistical Analysis of Asbestos Fiber Counting in the Laboratory & Industrial Environment" J. Am. Ind. Hygiene Assoc. pp. 909-915 (Dec. 1975)

3. General:
   a. Memorandum MESA 12/13/74
   b. Field Information Memorandum OSHA #74-92 11/21/74
   c. Procedure for Fiber Counting by F. G. Serafin 2/23/76

Julie C. Yang

JCY:mlr

attachments

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From: J. C. Yang
12/9/76

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TABLE I

EFFECT OF DIFFERENT VIEWING EQUIPMENT ON REPRODUCIBILITY OF COUNTING BY A SINGLE OPERATOR

(Reference: T&A 49561-2 Operator: J. Foley)

A)

Sample No.	TV	Microscope	MEAN ($\bar{X}$)	VARIATION $\lvert Xi - \bar{X} \rvert$	VARIABILITY %
1	4.31	3.8	4.055	0.255	6.29
2.	0.11	< 0.11	0.11	0	0
3.	0.68	1.20	0.94	0.26	27.66
4.	2.17	2.62	2.395	0.225	9.39
5.	3.48	3.76	3.62	0.14	3.87
6.	1.82	1.94	1.88	0.06	3.19
7.	0.17	0.17	0.17	0	0
8.	4.72	3.75	4.235	0.485	11.45
9.	0.14	0.29	0.215	0.075	34.88
10.	0.04	0.04	0.04	0	0
11.	1.51	0.88	1.195	0.35	26.36
				av:	11.19%

(Reference: T&A 49431 Operator: J.P.Wallace)

B)

Sample No.	TV	Microscope	$(\bar{X})$	$\lvert Xi - \bar{X} \rvert$	%
1.	2.64	4.08	3.36	0.72	21.43
2.	2.53	3.29	2.91	0.38	13.06
3.	2.61	3.25	2.93	0.32	10.92
4.	3.42	4.10	3.76	0.34	9.04
5.	5.24	3.87	4.555	0.685	15.04
6.	3.19	2.96	3.075	0.115	3.74
7.	2.28	2.10	2.19	0.09	4.10
8.	2.61	3.69	3.15	0.54	17.14
9.	3.23	3.42	3.325	0.095	2.86
10.	1.67	2.58	2.125	0.455	21.41
11.	4.20	2.91	3.555	0.645	18.14
12.	3.94	4.10	4.04	0.10	2.48
13.	1.77	4.30	3.035	1.265	41.68
				av:	13.93%

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From: J. C. Yang
12/9/76

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TABLE 2

EFFECT OF DIFFERENT OPERATORS ON REPRODUCIBILITY USING THE SAME VIEWING EQUIPMENT

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(Reference: T&A 49561-2)

A) Microscope Viewing

Sample No.	Operator 1	Operator 2	MEAN ($\bar{X}$)	VARIATION $\lvert Xi - \bar{X} \rvert$	VARIABILITY %
1	3.80	3.08	3.44	0.36	10.47
2	<0.11	<0.11	0.11	0	0
3	1.20	0.68	0.94	0.26	27.66
4	2.62	1.60	2.11	0.51	24.17
5	3.76	3.36	3.56	0.2	5.62
6	1.94	1.94	1.94	0	0
7	0.17	0.17	0.17	0	0
8	3.75	4.23	3.99	0.24	6.01
9	0.29	0.14	0.215	0.075	34.88
10	0.04	0.09	0.065	0.025	38.46
11	0.88	0.98	0.93	0.05	5.38
				av.	12.23%

(Reference: T&A 49431)

B)

Sample No.	Operator 1	Operator 2	($\bar{X}$)	$\lvert Xi - \bar{X} \rvert$	%
1	3.12	4.08	3.6	0.48	13.3
2	3.04	3.29	3.17	0.12	3.79
3	3.26	3.25	3.26	0	0
4	4.10	4.10	4.10	0	0
5	3.65	3.87	3.76	0.11	2.93
6	2.74	2.96	2.85	0.11	3.86
7	2.46	2.10	2.28	0.18	7.89
8	2.17	3.69	2.43	1.27	52.26
9	3.42	3.42	3.42	0	0
10	2.28	2.58	2.43	0,15	6.17
11	4.74	2.91	3.58	0.67	18.72
12	3.11	4.14	3.63	0.51	14.05
13	2.79	4.30	3.55	0.75	21.13
				av.	11.08%

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From: J. C. Yang
12/9/76

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TABLE 2 (continued)

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B) TV Screen Viewing

a) (Reference: T&A 49431)

Sample No.	Operator 1	Operator 2	MEAN ($\bar{X}$)	$\lvert Xi - \bar{X} \rvert$	%
1	1.68	2.64	2.16	0.48	22.22
2	3.80	2.53	3.17	0.635	20.06
3	3.91	2.61	3.26	0.65	19.94
4	3.65	3.42	3.54	0.115	3.25
5	3.65	5.24	4.45	0.795	17.87
6	1.25	3.19	2.22	0.97	43.69
7	2.46	2.28	2.37	0.09	3.80
8	3.26	2.61	2.94	0.325	11.07
9	3.61	3.23	3.42	0.19	5.56
10	2.13	1.67	1.90	0.23	12.10
11	3.28	4.20	3.74	0.46	12.23
12	2.28	3.94	3.11	0.83	26.69
13	1.39	1.77	1.58	0.19	12.03
				av:	14.49%

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From: J. C. Yang
12/9/76

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TABLE 2 (continued)

B)

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b) (Reference: T&A 49930)

Sample No.	Operator 1	Operator 2	MEAN ($\bar{X}$)	$\lvert Xi - \bar{X} \rvert$	%
1	1.32	1.64	1.48	0.16	10.81
3	3.70	3.49	3.595	0.105	29.17
4	0.90	0.77	0.835	0.065	7.78
5	0.91	1.00	0.955	0.045	4.71
6	0.54	0.54	0.54	0	0
7	1.16	1.42	1.29	0.13	10.07
8	0.99	1.16	1.075	0.085	7.91
9	1.17	1.15	1.16	0.01	0.86
10	0.96	1.03	0.995	0.035	3.52
11	1.43	1.18	1.305	0.125	9.58
12	0.22	0.33	0.275	0.055	20.0
18	0.42	0.54	0.48	0.06	12.5
19	0.19	0.25	0.22	0.03	13.64
20	0.02	0.05	0.035	0.015	42.86
21	0.34	0.24	0.029	0.005	17.24
22	0.31	0.26	0.285	0.025	8.77
23	0.42	0.36	0.39	0.03	7.69
24	0.40	0.38	0.39	0.01	2.56
25	0.15	0.20	0.175	0.025	14.29
26	0.28	0.28	0.28	0	0
				av:	11.20%

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From: J. C. Yang
12/9/76

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TABLE 3

EFFECT ON REPRODUCIBILITY USING THE SAME EQUIPMENT AND THE SAME OPERATOR

03630487

(Reference: T&A 49930, Operator: J.Foley)

A) Microscope Viewing

Sample No.	Trial I	Trial II	$\bar{X}$	$\lvert Xi - \bar{X} \rvert$	%
1	2.02	1.58	1.9	0.22	11.58
3	4.95	5.32	5.135	0.185	3.60
4	1.26	1.30	1.28	0.02	1.56
5	1.43	1.00	1.215	0.215	17.70
6	0.83	0.81	0.82	0.01	1.22
7	1.80	1.53	1.67	0.14	8.38
8	1.42	1.46	1.44	0.02	1.39
9	1.55	1.53	1.54	0.01	0.65
10	0.75	0.68	0.715	0.035	4.90
11	1.51	2.07	1.79	0.28	15.64
12	0.27	0.31	0.29	0.02	6.9
18	0.37	0.58	0.475	0.105	22.11
19	0.27	0.29	0.28	0.01	3.58
20	0.04	0.02	0.03	0.01	50.0
21	0.35	0.28	0.33	0.05	15.15
22	0.35	0.29	0.32	0.03	9.37
23	0.48	0.50	0.49	0.09	18.37
24	0.26	0.20	0.23	0.03	13.04
25	0.17	0.12	0.145	0.025	17.24
26	0.20	0.20	0.20	0	0
				av:	8.87%

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From: J. C. Yang
12/9/76

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TABLE 3 (continued)

EFFECT OF REPRODUCIBILITY USING THE
SAME EQUIPMENT AND THE SAME OPERATOR

(Reference: T&A 49930, Operator: J.Foley)

03630488

B) TV Screen Viewing

Sample No.	Trial I	Trial II	$\bar{X}$	$\lvert Xi - \bar{X} \rvert$	%
1	1.32	1.73	1.52	0.20	13.16
3	3.70	5.27	4.485	0.785	17.50
4	0.90	0.73	0.815	0.088	10.452
5	0.91	1.18	1.045	0.135	12.92
6	0.54	0.76	0.65	0.11	16.93
7	1.16	1.46	1.31	0.15	11.45
8	0.99	1.16	1.075	0.085	7.91
9	1.17	1.03	1.1	0.07	6.36
10	0.96	0.83	0.895	0.065	7.26
11	1.43	1.77	1.6	0.17	10.63
12	0.22	0.40	0.31	0.09	29.03
18	0.42	0.50	0.46	0.04	8.70
19	0.19	0.29	0.24	0.05	2.08
20	0.02	0.02	0.02	0	0
21	0.34	0.28	0.31	0.03	9.68
22	0.31	0.20	0.255	0.055	21.57
23	0.40	0.36	0.39	0.03	7.69
24	0.40	0.28	0.34	0.06	17.65
25	0.15	0.12	0.135	0.015	11.11
26	0.28	0.33	0.305	0.025	8.20
				av:	12.06%

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From: J. C. Yang
12/9/76

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TABLE 4

SUMMARIZED RESULTS OF TABLES 1,2 & 3

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VARIABLES	CONSTANTS	% VARIABILITY	Ave.
Viewing equipment	Operator	11.19	10.85
Viewing equipment	Operator	13.93	
Viewing equipment	Operator	7.42	
Operator	Viewing Equipment-Microscope	12.23	11.66
Operator	Viewing Equipment-Microscope	11.08	
Operator	Viewing Equipment - TV	14.49	12.85
Operator	Viewing Equipment - TV	11.20	
Repeats, viewing fields	Viewing Equipment, operator - Misc.	8.87	
	Viewing Equipment, operator - TV	12.06	
	Average Total Variability	11.39%	

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108Z00230

From: J. C. Yang
12/9/76

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TABLE 5

STATISTICAL STUDY OF COUNTING DATA
OF LIBBY SAMPLE AT CAMBRIDGE
(average of 6 countings *)

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Sample No.	Mean	Variance	Standard Deviation	Standard Error of Arith. Mean
1	1.64	0.05	0.23	0.095
3	4.51	0.63	0.79	0.324
4	0.99	0.06	0.24	0.099
5	1.06	0.06	0.22	0.089
6	0.67	0.05	0.14	0.057
7	1.45	0.02	0.22	0.088
8	1.19	0.05	0.22	0.088
9	1.24	0.05	0.24	0.097
10	0.88	0.06	0.14	0.06
11	1.03	0.02	0.68	0.216
12	0.31	0.47	0.06	0.025
18	0.49	0.004	0.08	0.032
19	0.24	0.006	0.05	0.021
20	0.03	0.003	0.01	0.005
21	0.29	0.0002	0.06	0.023
22	0.28	0.003	0.05	0.021
23	0.42	0.004	0.06	0.025
24	0.30	0.006	0.08	0.031
25	0.15	0.001	0.03	0.012
26	0.25	0.003	0.05	0.021
	av:	.075	0.18	

*Reference (Table 1C, 2Bb, 3A and 3B)

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TABLE 6

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COMPARISON OF LIBBY COUNTING DATA
WITH CAMBRIDGE COUNTING DATA

(Reference: T&A 49930)

Sample No.	Libby	Cambridge*	HE**	FGS†	Libby on Cam slides
1	3.85	1.64	2.75		2.22
3	8.43	4.51	5.93	5.37	4.89
4	2.07	0.99			1.2
5	2.50	1.06	1.62		1.2
6	1.86	0.67			2.0
7	2.82	1.45			1.31
8	2.48	1.19			1.59
9	2.72	1.24			1.93
10	2.70	0.88			1.15
11	2.78	1.03			1.63
12	0.63	0.31			
18	1.89	0.49			
19	0.84	0.24			
20	0.47	0.03			
21	0.76	0.29			
22	0.78	0.28			
23	1.59	0.42			
24	0.93	0.30			
25	0.69	0.15			
26	1.20	0.25		0.30	

*The Cambridge data is the mean of 6 countings made by 2 operators, 3 on TV and 3 on microscope. The values were different from those shown in T&A 49930 in which samples 1, 3, 4, 5 were average of 4 countings, and the remainder were average of 2 values; subsequently, more countings were made after the report was issued. Also, value for sample 1 in T&A 49930 should be 1.66 instead of 2.66 (written error).

**HE - Counting made by H. Eschenbach

†FGS - Counting made by F. G. Serafin

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Construction Products Division

03630492

TO: H. C. Duecker
H. A. Eschenbach
R. H. Locke
J. W. Wolter
R. L. Oliverio/CPD Libby
File: 71-046

DATE: December 14, 1976

SUBJECT: Correction for T&A 49930

FROM: J. E. Foley

cc: J. C. Yang

The correct fiber count for sample 10-4-76-1 should be 1.66 (average value of 4 separate counts - namely, 1.32; 1.73; 2.02 and 1.58) instead of 2.66, a written error.

J. E. Foley

JEF:mlr

J E Foley

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108Z00233

486355

ADMINISTRATIVE RECORD

NUMBER: 49189
GROUP: ZONOLITE
ACTUAL COST: $2500.00
REPORTING DATE: May 26, 1977

REQUEST FOR TECHNICAL SERVICE:

CONFIDENTIAL

03627765

SUMMARY:

Three bags of standard MK-4 product from plant locations in California from Los Angeles, Santa Ana, and Newark), and four MK-5 samples (from Los Angeles, Santa Ana, and Omaha) have been examined for their tremolite content.

All seven samples as received showed no detectable tremolite fiber content by x-ray determinations (our detection limit for tremolite is 0.2%). However, the materials were fractioned; glass fibers were mostly retained on a +6 mesh screen, vermiculite was floated off; most of the plaster of Paris was dissolved in water; and, CELIF fibers and organic matter were burnt off. The concentrated fines, collected on Millipore filter of 0.45 μ, showed the presence of trace amounts of tremolite fiber in two of the trace MK-4 samples (Santa Ana and Newark). By petrographic microscopic examination, this was estimated to be less than 0.015% of the total sample.

The concentrates were then submitted to Arthur D. Little, Inc., for transmission and scanning electron microscopic analysis (TEM and SEM), selected area electron diffraction (SAED) and energy dispersive x-ray analysis (EDAX).

By these sophisticated and time-consuming instrumental analyses, the amphibole fibers were positively identified and analyzed. On a mass basis, it was found to be less than 0.006% of the concentrates which corresponded to 1.7 ppm* (Santa Ana) and 4.1 ppm (Newark) of the total MONOKOTE® sample weight.

EXPERIMENTAL:

Concentration

The concentration procedure of MONOKOTE is shown in Figure 1. The results are tabulated as follows:

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* parts per million, or 0.00017%.

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068ETX02089

REQUEST FOR TECHNICAL SERVICE

NUMBER:	49189
GROUP:	ZONOLITE
ACTUAL COST:	$2500.00
REPORTING DATE:	May 26, 1977

Fraction	Description	Material Present	% by weight in each Fraction						
			MK-4			MK-5			
			L.A. (8/76)	S.A. (8/76)	Newark (8/76)	Omaha new (8/76)	Omaha old	S.A. (10/76)	L.A. (10/76
1	Soluble	plaster of Paris	28.5	40.2	46.0	33.3	37.2	43.6	40.4
2	+6 Mesh	glass fiber	57.8	56.9	47.1	56.0	49.2	49.1	55.3
3	-6 +50	glass fiber, expanded Vm. some insoluble plaster							
4	Fines	some insoluble plaster, fine Vm. and tremolite (?), gypsum	13.7	2.9	6.9	10.7	13.6	7.3	4.3
			100.0%	100.0	100.0	100.0	100.0	100.0	100.0

X-Ray Diffraction Analysis

No detectable tremolite found in any of the fractions of the seven samples.

REQUEST FOR TECHNICAL SERVICE:

CONFIDENTIAL

NUMBER:	49189
GROUP:	ZONOLITE
ACTUAL COST:	$2500.00
REPORTING DATE:	May 26, 1977

Petrographic Microscopic Examination

03627767

Based on the characteristic refractive indices and optical properties of vermiculite and tremolite fibers, using the liquid immersion technique, a trace of tremolite was found in the -50M +0.45 μ portion of Santa Ana MK-4, and Newark MK-4 samples.

Analysis by Arthur D. Little, Inc.

Even though the original request made by R. H. Locke was on one MK-4 and one MK-5 sample, we have decided to do several more since the product from each plant looked and behaved very differently. The MK-4 from Newark was very dense and the vermiculite present was poorly expanded in comparison with the others. Product from Santa Ana was very bulky and the plaster of Paris present in the composition dissolved more readily than the others.

The two concentrated samples suspected to have tremolite fibers were submitted to Arthur D. Little for fiber characterization and counting on transmission micrographs (Figures 2 and 3). Each fiber being counted was analyzed by SAED (selected area electron diffraction) to determine the structure of the fiber. It was found that 25-40% of fibers did not yield an SAED pattern indicating the fiber was amorphous, mostly organic and glass fibers. The breakdown of the fiber types and amounts is listed in Table 1.

Scanning electron micrographs were also taken on some of the fibers. They are shown in Figures 4 and 5, and energy dispersive x-ray analysis (EDAX) was employed to analyze the elements present in each fiber. The results are shown in Table 2.

CONCLUSIONS and COMMENTS:

The conclusion reached by A. D. Little, Inc. was that the amphibole fiber content, on a mass basis, corresponded to less than 0.006% of the supplied concentrated sample. Letter from Dr. E. Peters of ADL is attached. Computing the amphibole content in the MONOKOTE samples from Santa Ana and Newark, this corresponds to less than 1.7 ppm and 4.1 ppm, respectively. The level of tremolite fiber present was extremely low.

Julie C. Yang

JCY:mlr

attachment

20152835

068ETX02091

REQUEST FOR TECHNICAL SERVICE:

CONFIDENTIAL

NUMBER: 49189
GROUP: ZONOLITE
ACTUAL COST: $2500.00
REPORTING DATE: May 26, 1977

TABLE 1 - Fiber analysis by TEM (A.D.Little)

03627768

Fiber Observed	Sample 22281-1 Fines Fraction from Santa Ana, MK-4 Sample	Sample 22281-2 Fines Fraction from Newark, MK-4 Sample
Total fibers observed	104	54
% Amphibole	6	4
% Other Mineral (mostly gypsum)	33.5	35
% Ambiguous Mineral (with insufficient data for positive identification)	34.5	22
% Amorphous (organic, glass fiber)	26	39
	100%	100%

TABLE 2 - EDAX Microchemical Analysis of Fibers Observed by Scanning Electron Microscopy (A.D.Little)

		Relative X-ray Intensity			
Sample 22281-1		Strong	Medium	Weak	Probable I.D.
Fiber 1	Figure 6a	Al	S	Mg	
Fiber 2	Figure 6b	Si,Al	Mg,Ca,S	Fe,K	amphibole or glass
Fiber 3	Figure 4a	Al	-	Ca,S,Si	gypsum (?)
Fiber 4	Figure 4b	Si,Al,Mg,S	Ca,Fe	K	amphibole or glass
Sample 22281-2					
Fiber 5	Figure 3	S,Ca,Al			gypsum

20152836

GRACE

Construction Products Division

CHARACTERIZATION AND PREPARATION OF RESPIRABLE SIZED TREMOLITE FIBER AND VERMICULITE FOR ANIMAL STUDIES

by: Julie C. Yang

April 8, 1976

CAMBRIDGE

03627778

TO: H. C. Duecker

DATE: April 8, 1976

FROM: Julie C. Yang

SUBJECT: Characterization and Preparation of Respirable Sized Tremolite Fiber and Vermiculite for Animal Studies

CC: H. A. Brown
J. W. Wolter
H. A. Eschenbach
R. H. Locke
File: 71-048

PURPOSE

The objectives of this study are to find out the size distribution and concentration of the respirable size fibers and vermiculite on the air filter collected by the the Industrial Hygiene and Environmental Health group in the field, and to prepare the samples corresponding as closely as possible to these air filter material, for animal studies.

AIR FILTER STUDY

Several randomly collected air samples from Libby at fairly long time intervals were collected for fiber contents and submitted to Arthur D. Little for sizing and distribution studies.

Two samples were sent:

Sample No.	Collecting Time	Fiber Count (Optical/40 Fields)
22260P-1	248 mins.	0.18 Fiber/cc air
22260P-2	300 mins.	2.15 Fiber/cc air

The results from Arthur D. Little are shown in Tables 1 and 2, Figures 1 - 3; and conclusions reached are summarized as follows:

1) On the air filter the respirable sized vermiculites and tremolite fibers are roughly in 50-50% ratio.
2) The respirable size tremolite fibers are mostly less than 10 microns (< 8% > 10 μ size), and the geometric mean length of the fibers is around 3.1 μ.
3) The respirable size vermiculites are also less than 10 μ, having an average size about 5 μ.
4) The aspect ratio of the fibers is in the range of 11 to 15 μ.
5) Computation shows that the fiber counting with SEM (scanning electron microscope) @ 20,000 magnification. The total numbers of fibers found per unit area (1 cm^2) is about seven times in number of the fibers found by optical microscope counting at 400 magnification.

SEM shows 7X PCM

20152846

068ETX02102

To: H. C. Duecker
From: J. C. Yang

Re: Animal Studies
April 9, 1976

SAMPLE PREPARATIONS

After we characterized what we have on the air filter, attempts were made to prepare both respirable sized vermiculite and tremolite fibers as closely as possible to those found on the air filter.

From previous research work (report on Libby Ore Evaluation - Ore Impurities, 2/23/76) we have found that Libby #2 vermiculite product has the highest tremolite fiber content in the order of 5% by weight. Since the sizes of #2 are fairly and easily to be handpicked, it is used as a starting source for both tremolite and vermiculite.

The tremolite fiber bundles picked out from Libby #2 are fairly clean and free of rocks, greyish in color, soft, and sometimes waxy in touch. They broke down easily to fine fibrils when degraded, which looked extremely similar to those found on the filter or floating in air in the Libby operation, which are quite different than the tremolite found in associated veins in rock form; they are generally harder and harsher, most of which were removed in the floatation process.

1) Tremolite Fiber

a) Cleaning

Tremolite fiber bundles were hand-picked from Libby #2 product, cleaned with acetone and then distilled water. The bundles were then opened with Waring Blender for 2 minutes at high speed, filtered and dried in the oven at 105°C. for about four hours.

b) Milling

The oven-dried material was Spec-milled in 0.5 g batch for a total of 45 seconds; but after each 10 seconds milling interval the mill was stopped and the material reruffled to avoid excessive packing.

The Spec-milled samples were then chilled in dry ice-acetone batch, chilling at low temperature increases the brittleness of the fibers and makes them easier to be pulverized. The chilled fibers were subjected to a Wiley mill with a built-in 60 mesh screen, a mill which has been designed especially for milling fibers. The Wiley milling was repeated another three times. Between runs the material has to be chilled again thoroughly with dry ice.

c) Sedimentation

0.8 g of the Wiley milled sample (mostly 2-4 μ in size, some up to 30 μ with some bundles under light microscope) was dispersed in two liters of distilled water, allowed to stand for 20 minutes; then, decant the cloudy solution into 250 ml or 500 ml graduated cylinders which were employed as sedimentation columns, and dilute the solution to twice its volume with distilled water. The solutions in each column were lightly stirred and allowed to settle for twenty minutes. The cloudy solution was then filtered by an HA type Millipore filter of 0.45 μ. However, the filterate looked extremely clear and showed some small particles under the microscope.

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From: J. C. Yang

Re: Animal Studies
April 9, 1976

The solid collected from the beaker and the column were recombined and treated with another 2 liters of distilled water, poured into columns and allowed to stand overnight. The cloudy solution was again decanted and filtered through the Millipore. Coarse solid remained at the bottom of the column from the second sedimentation, was filtered and saved for future remilling. The five fibers collected on the top of the Millipore were then examined by light microscope. It was found most of the particles were around 2 μ, and a few long fibers up to 20 μ.

d) Cleaning and Resizing

The finished crude product from step c. was redispersed in the order of 2 g/4 liter distilled water, and allowed to stand in columns for over half an hour. The decanted cloudy solution (about twice as dense as solution in step c.) was then filtered through Millipore filter. The solid left at the bottom of the column was dispersed again, ultrasonically, for 2 minutes in 400 ml water. The milky solution was then diluted to another 4 liters and allowed to settle in columns for a final 20 minutes. The fines were collected on Millipore by filtering the decanted liquid, dried as examined by light microscope. The product has mostly 2 μ in size, very few larger fibers but a few up to 10 μ. The solid remained from decantation was again filtered and saved for future remilling.

2) Vermiculite

a. Cleaning

The vermiculite platlets were also hand-picked from Libby #2 product, cleaned in Soxhlet extractor with isopropyl alcohol, then acetone, and finally water to remove all the trace of organic contaminants used in the flotation process; then oven-dried at 105°C. for several hours.

b. Milling

The oven-dried vermiculite was then chilled with acetone and dry-ice mixture, Spec-milled in 2 g batches for 10 minutes. At the end of 5 minutes, the mill was stopped and the material was reruffled.

c. Screening

The milled sample was screened with 325 mesh screen. The -325 mesh product showed the desirable respirable size. Most of the particles were 2 - 4 μ. Some large plates were about 10 - 15 μ. The +325 mesh material was also collected and saved for future remilling.

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From: J. C. Yang

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April 9, 1976

3) Proportioning

5 g of tremolite and 5 g of vermiculite, prepared from step 1) and 2) respectively, were carefully weighed out on a semimicro balance, and then transferred to a 4 oz. size wide-mouth glass bottle in which some silver wires were added to break up the powder surface when mixed on a roller mill. The mixing was carried out for about 16 hours. Because of the morphology and density difference, it will be suggested to Dr. Smith that when this sample is being used for animal study, an appreciable quantity (such as 1 or 2 grams) is taken, then dispersed in the saline medium ultrasonically, prior to use. The purpose of doing this will eliminate the localized inhomogenity and selectiveness of a very small sample.

4) Characterization

The respirable-sized fibers (2260P-4 and 22250P-5) have been sent to A. D. Little for sizing and comparison with the fiber found on the air filter. The results are also shown in Tables 1 and 2, Figures 7 and 8. Scanning electron micrographs of these materials are shown in Figures 9 - 10.

Results from A. D. Little and our own microscopic sizing indicated that the respirable size fibers and vermiculite which we prepared are very similar to those on the air filter. However, sample 22260P-4 is a fiber sample of finer size, extremely time-consuming to obtain in large quantities. We have then taken a different approach to obtain 22260P-5 which is slightly coarser than 22260P-4. The two samples of 8 grams each we have submitted to Dr. W. Smith are:

1. 22260P-5 - respirable sized tremolite fiber
2. 22263P-2 - a mixture in 50-50% of respirable sized tremolite fiber (22260P-5) and vermiculite (22263P-1)

The final characterization of samples will be made by Walter McCrone Associates:

1. 22260P-5 respirable sized tremolite fiber
2. 22263P-1 respirable sized vermiculite
3. 22263P-3 a saline suspension of 22263P-2 will be prepared by W. Smith's group for animal studies.

To: H. C. Duecker
From: J. C. Yang

Re: Animal Studies
April 9, 1976

03627782

5) Sample Preparation for Animal Injection Studies

Dr. Smith's group has been preparing samples by dispersing 2 g of the solid in 40 ml 0.9 g saline solution in a 100 ml Erlenmeyer flask, then autoclaved for 15 minutes at 15 - 20 psi to sterilize the material. After it was cooled off, the mixture was shaken by hand and drawn into a syringe in 1 ml aliquot for injection.

By observing the preparations made with R. T. Vanderbilt sample (talc and tremolite mixture), solid settled very quickly in the saline solution immediately after shaking. Employing such technique, I would expect the animals got different doses of material depending on the technique of the operator and the rate of settling at that specific time. In addition, the fibers present may be in bundles or small balls not fully opened.

As a result, I have recommended the use of ultrasonic dispersion. The saline suspension after autoclaved should be subjected to a 10 minute sonic dispersion. It has been demonstrated the respirable sized material was suspended quite uniformly for an hour or more without settling. In case of any fiber balls or bundles present, they will be fully opened and dispersed, too.

Each animal will get 1 ml of the suspension which has 25 mg of the solid theoretically.

Julie C. Yang

JCY:mlr

attachments

20152850

TABLE 1

SUMMARY OF LENGTH DATA

03627783

Range (μ)	No. 1 (Total No.) N	Cum %	No. 2 N	Cum %	22260-P4 N	Cum %	22260-P5 N	Cum %
<0.3	2	4	0	0	0	0	0	0
0.3-0.4	6	14	1	1	1	1	0	0
0.4-0.5	4	21	1	2	4	4	1	1
0.5-0.6	6	32	1	2	3	7	2	3
0.6-0.7	0	32	2	4	5	12	0	3
0.7-0.8	7	44	5	8	3	14	3	5
0.8-0.9	2	47	4	11	3	17	3	9
0.9-1.0	1	49	0	11	4	20	2	11
1.0-1.1	2	53	3	14	7	27	7	18
1.1-1.2	1	54	1	15	3	29	2	20
1.2-1.3	3	60	4	18	5	34	2	22
1.3-1.4	0	60	2	20	1	35	7	29
1.4-1.5	0	60	5	24	4	38	7	35
1.5-1.6	1	61	1	24	4	42	5	41
1.6-1.7	1	63	4	28	5	46	1	42
1.7-1.8	2	67	0	28	0	46	2	44
1.8-1.9	0	67	1	28	4	50	6	50
1.9-2.0	2	70	2	30	1	50	3	53
2.0-2.5	0	70	4	33	7	57	10	63
2.5-3.0	3	75	16	46	13	68	12	75
3.0-3.5	1	77	6	51	8	76	3	78
3.5-4.0	0	77	8	58	6	81	4	82
4.0-4.5	2	81	9	65	1	82	0	82
4.5-5.0	1	82	2	67	3	85	2	84
5.0-6.0	0	82	13	77	4	88	5	89
6.0-7.0	2	85	2	79	4	92	6	95
7.0-8.0	4	93	9	86	4	96	2	97
8.0-9.0	2	96	3	89	2	97	1	98
9.0-10.0	0	96	3	91	2	99	0	98
>10.0	2	100	11	100	1	100	2	100
Total	58		123		113		125	

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Arthur D Little Inc

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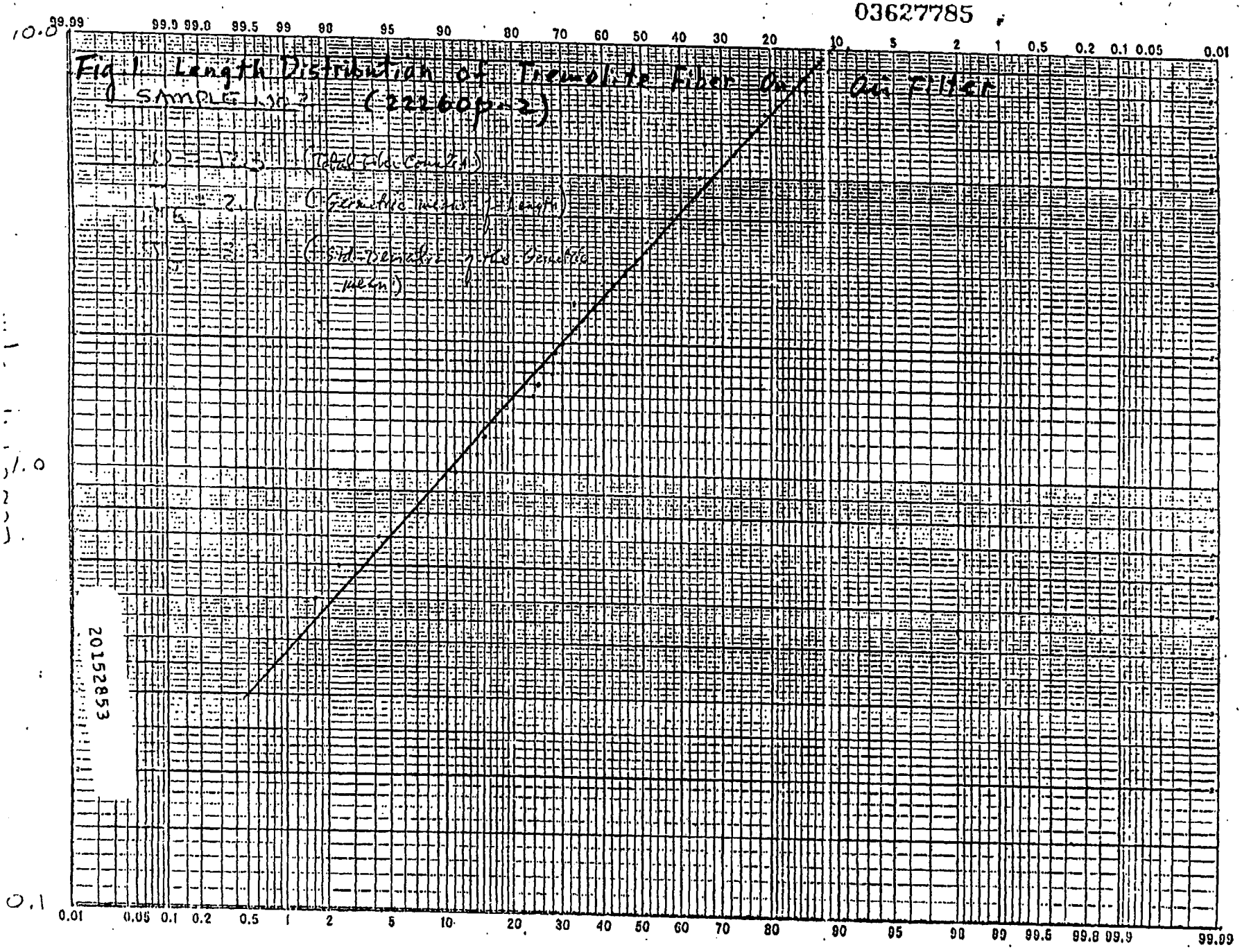
TABLE 2

SUMMARY DATA FROM A. D. LITTLE

Sample No.:	22260P-1	22260P-2	22260P-4	22260P-5
Total Fibers Counted	57	123	113	125
Arithmatic Means				
Length (μ)	2.59	4.34	2.76	2.79
Width (μ)	0.26	0.39	0.15	0.24
Average of Aspect Ratio	15.85	15.86	22.50	13.39
Mass (10^{-12}g)	0.5218	2.0464	0.1925	0.4982
Geometric Means				
Length (μ)	1.38	3.11	1.97	2.07
Std. Deviation/Avg. Length	6.6	3.5	2.4	2.0
Width (μ)	0.12	0.27	0.12	0.20
Average of Aspect Ratio	12.01	11.42	16.147	10.36
Mass (10^{-12}g)	0.0571	0.7162	0.0880	0.2584
Fibers/cm^2	52,660	295,430		
Fiber Mass/cm^2 (10^{-9} g)	27.5	606.4		

J.C.Yang 4/8/76

20152852



TREMOLITE FIBER BUNDLES
(Handpicked from Libby #2 Product)

CLEANING

Washing ↓ acetone / H_2O

Opening ↓ Waring Blender / hi speed / 2 mins

filtering ↓

drying ↓ oven / 105°C / 4 hrs

MILLING

Spec-milling ↓ 45 sec / reruffle sample every 10 sec.

Freeze ↓ ↑ dry ice / acetone

Wiley milling ↓ repeat 4 times

DISPERSION & SIZING

disperse ↓ 1g solid / 2.5 ℓ. dist. H_2O

stand ↓ 20 min

decant ↓

Solid

Cloudy Soln

dilute ↓ 2X / dist. H_2O

stand ↓ 20 min

decant ↓

Solid

Cloudy Soln

redisperse ↓ 1g / 2.5 ℓ. distilled H_2O

stand ↓ in columns 20 min or over

decant ↓

filter ↓ Millipore 0.45 μ

Solid (saved for future regrinding)

Cloudy liq.

filter ↓ Millipore, 0.45 μ

Crude Product

FIGURE 4 - TREMOLITE PREPARATION (Continued)

J.C. Yan
3/23/76

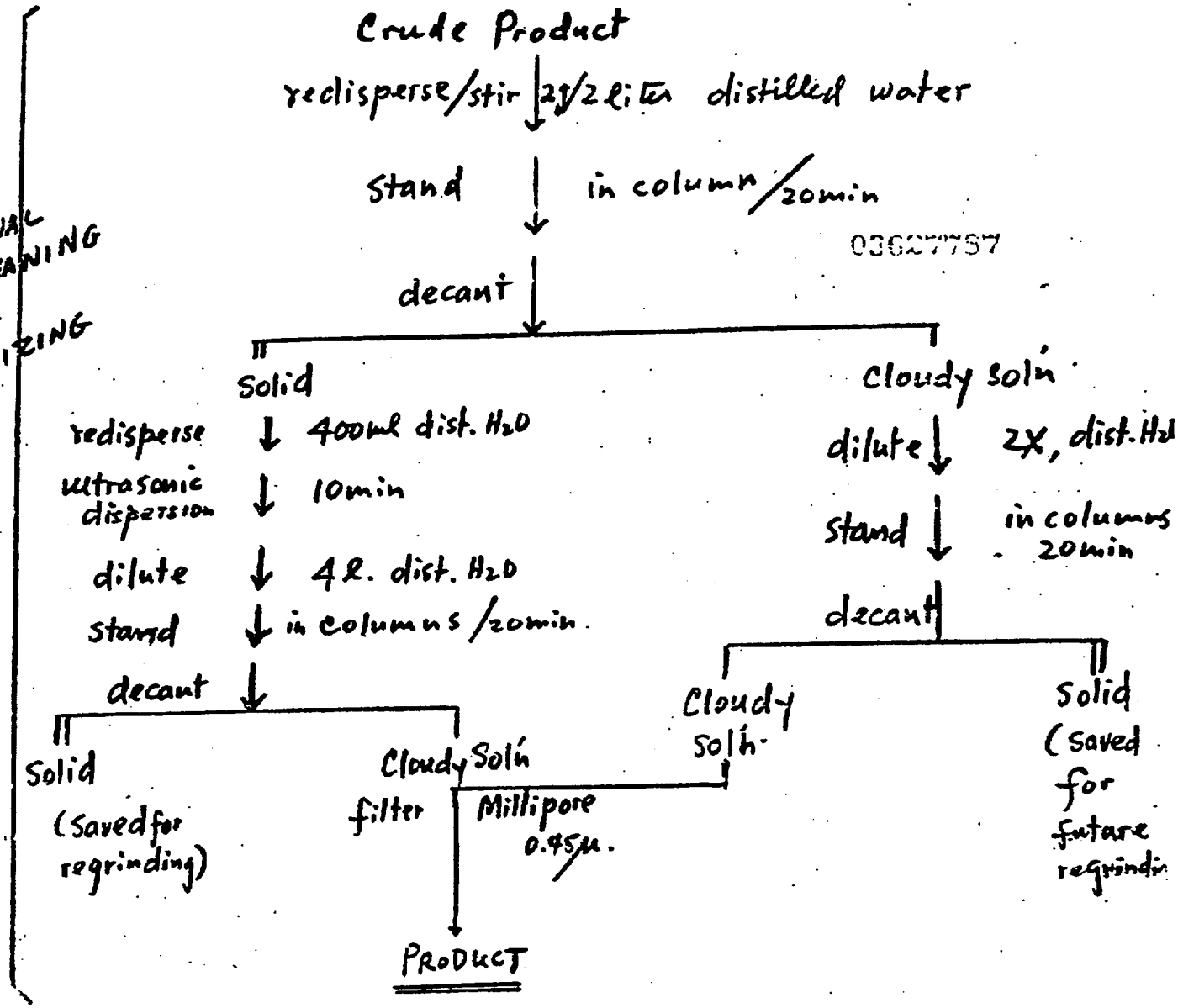


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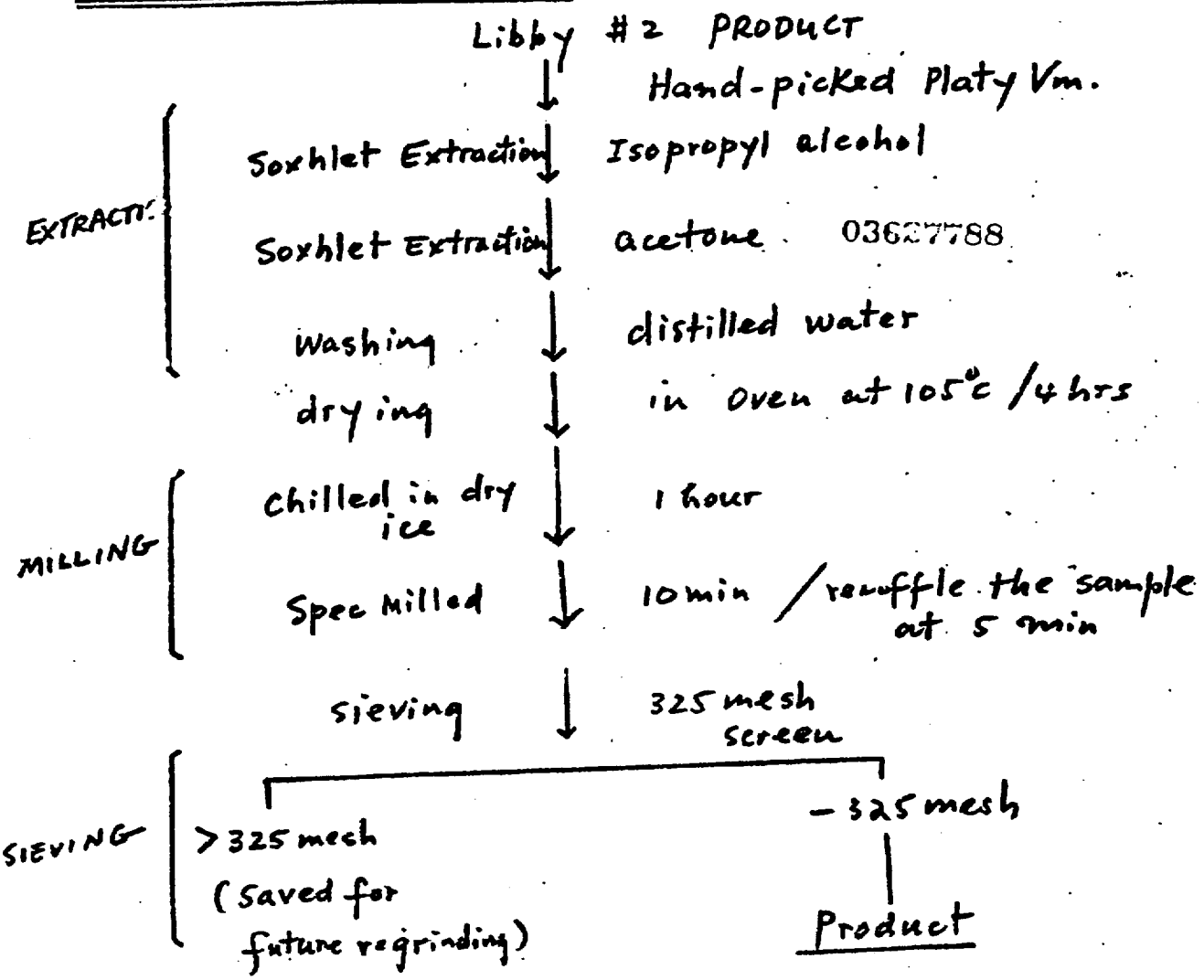
Fig. 5

VERMICULITE PREPARATION



J.C. Yang /3/24/.

Fig. 6
PROPORTIONING

03627789

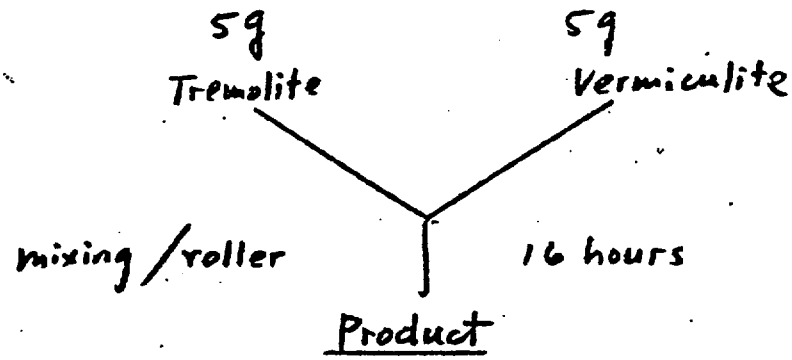


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Fig 7 Length Distribution of Tremolite Fiber Prepared (22260p-4)

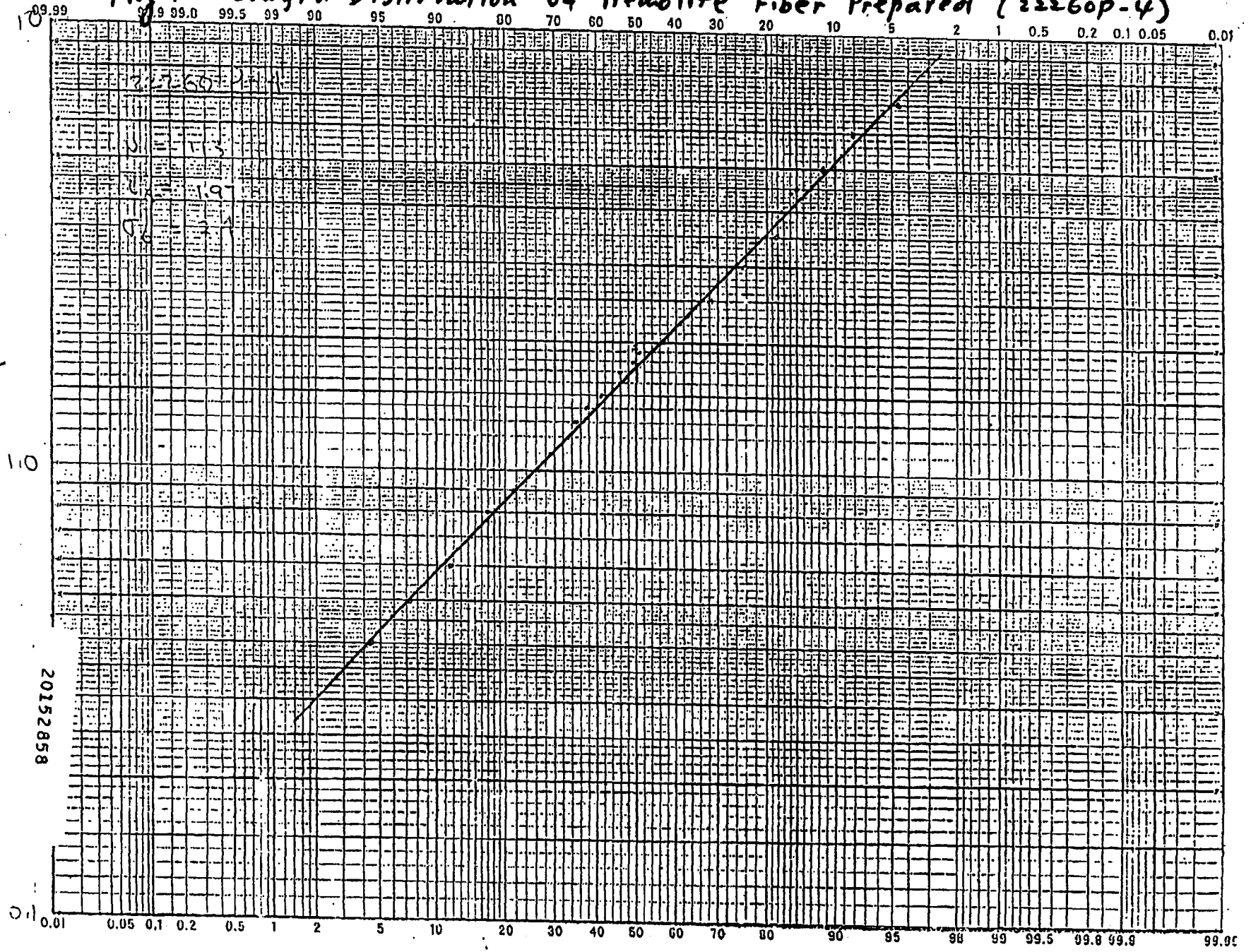


Fig 8 Length Distribution of Tremolite Fiber Prepared & Submitted To Dr. W. Smith (22360 P-5)

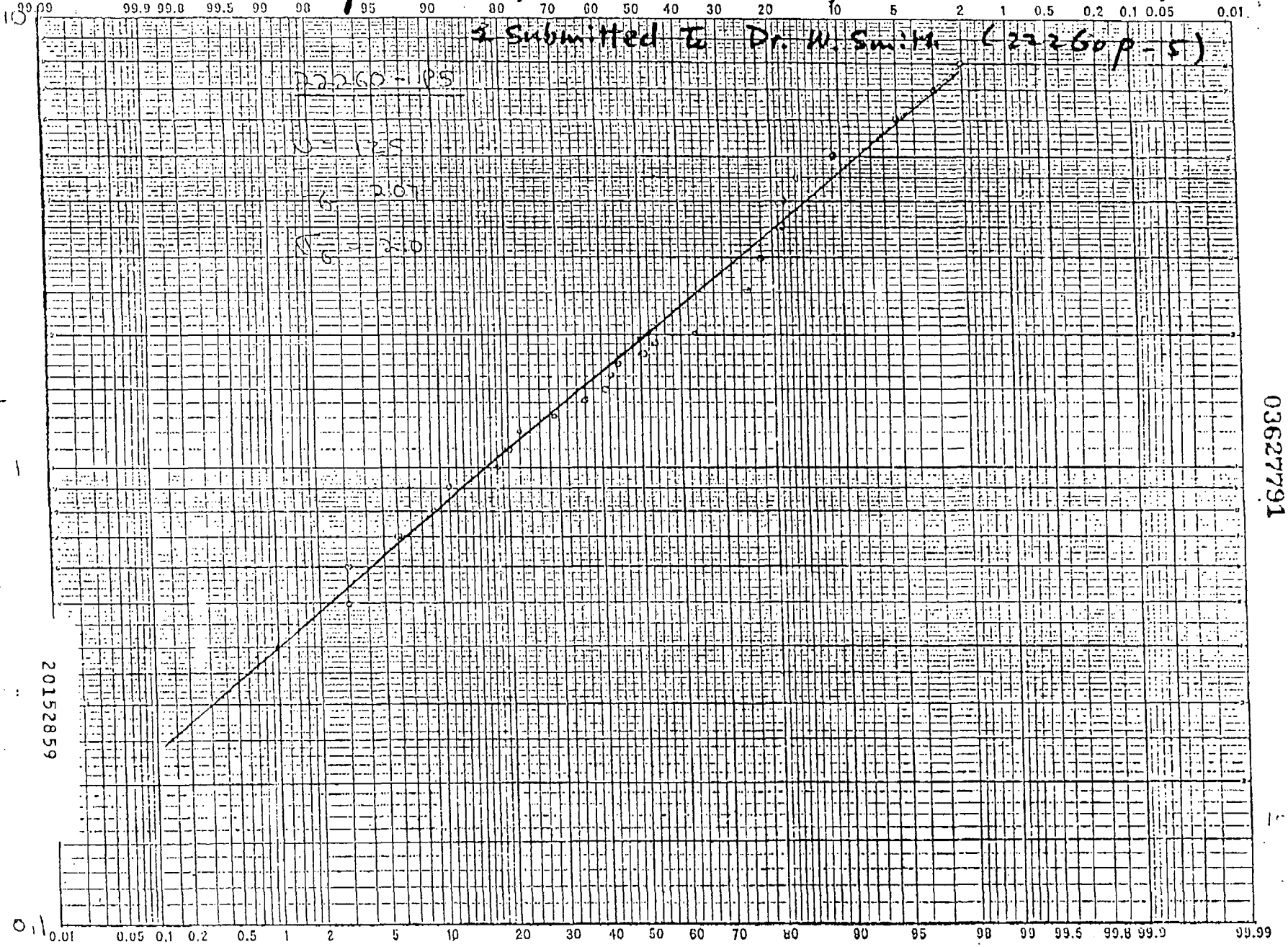
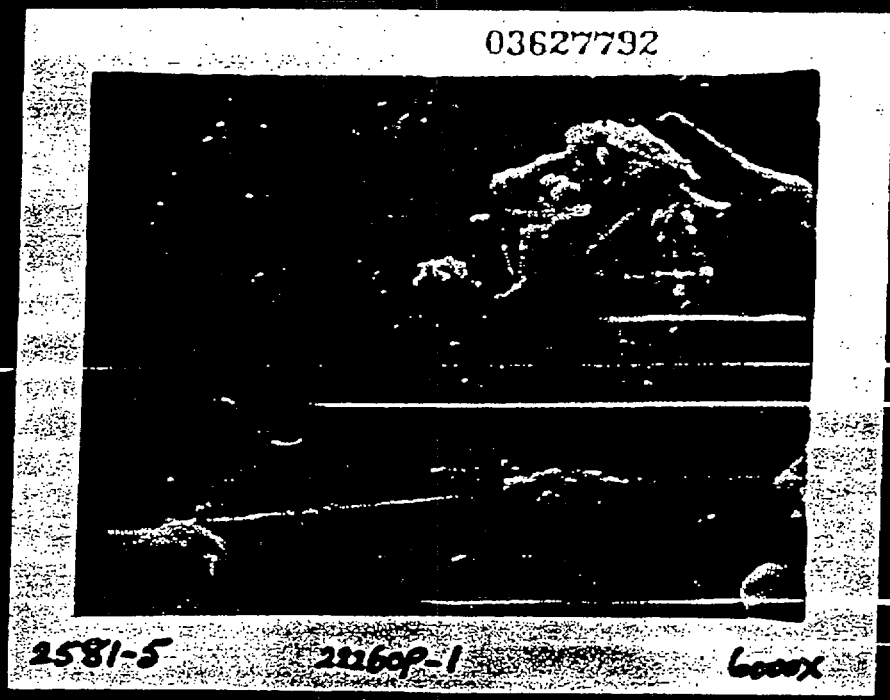


FIGURE 2
SCANNING ELECTRON MICROGRAPHS (SEM)
OF AIR FILTER #1



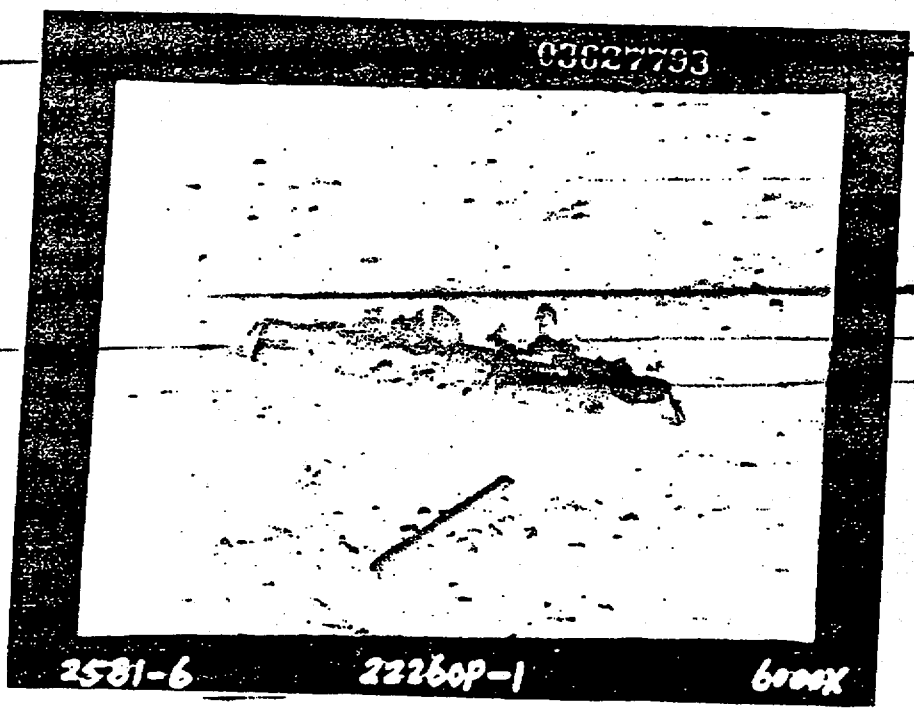


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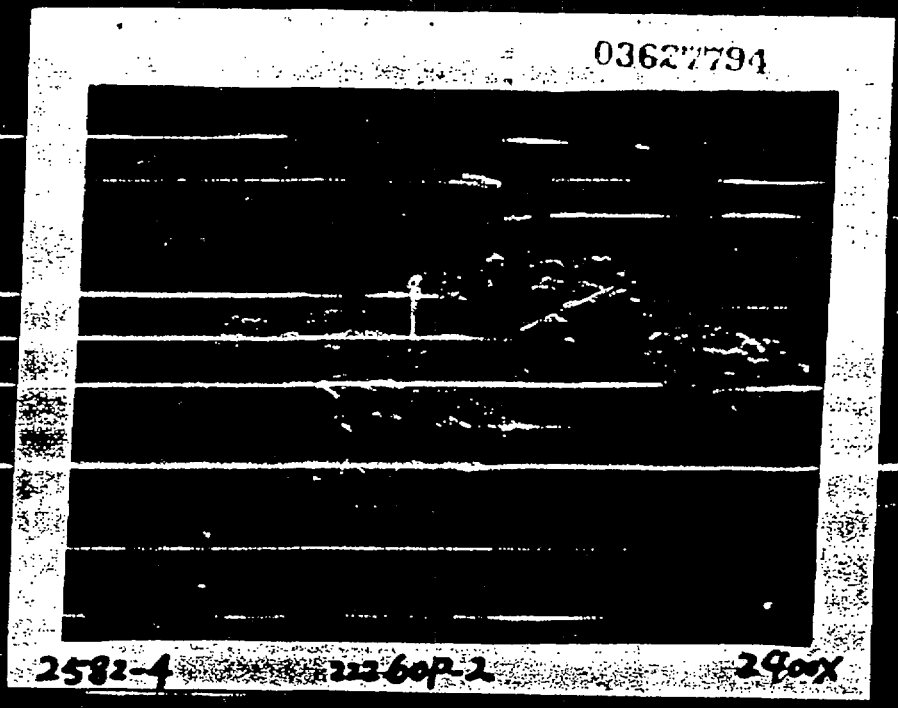
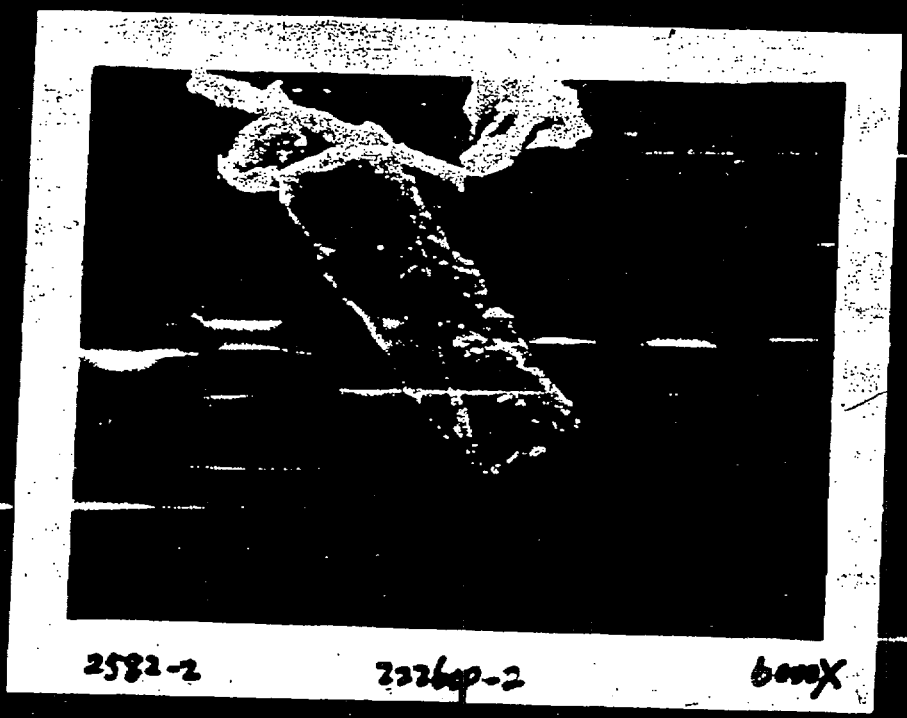
FIGURE 3
SEM OF AIR FILTER #2





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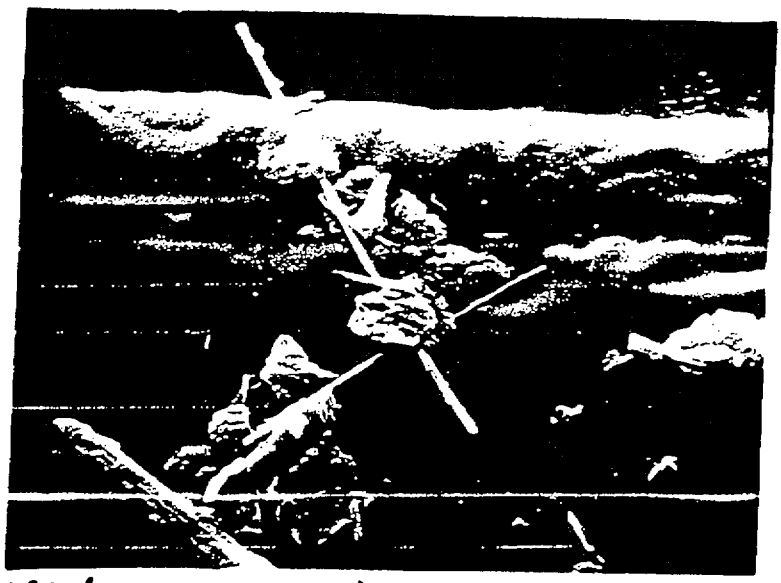
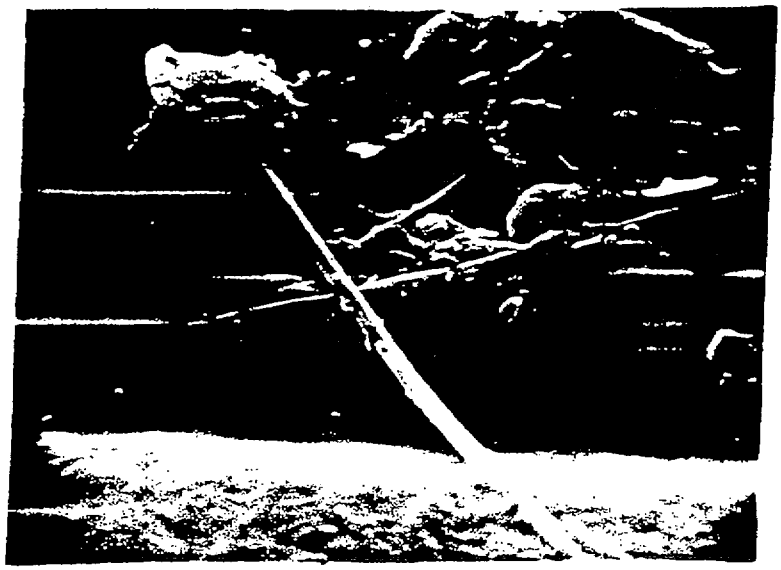


2582-6 22260P-2 2400X



2582-3 22260P-2 6000X

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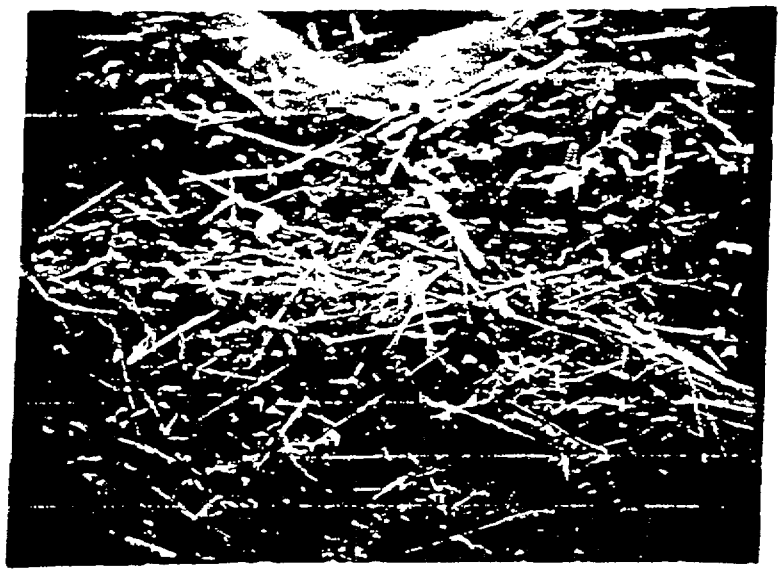
FIGURE 9

SEM OF RESPIRABLE SIZE TREMOLITE FIBER PREPARED (22260P-4)



2607-5 22260P-4 2400X

2607-1 22260P-4 6000X

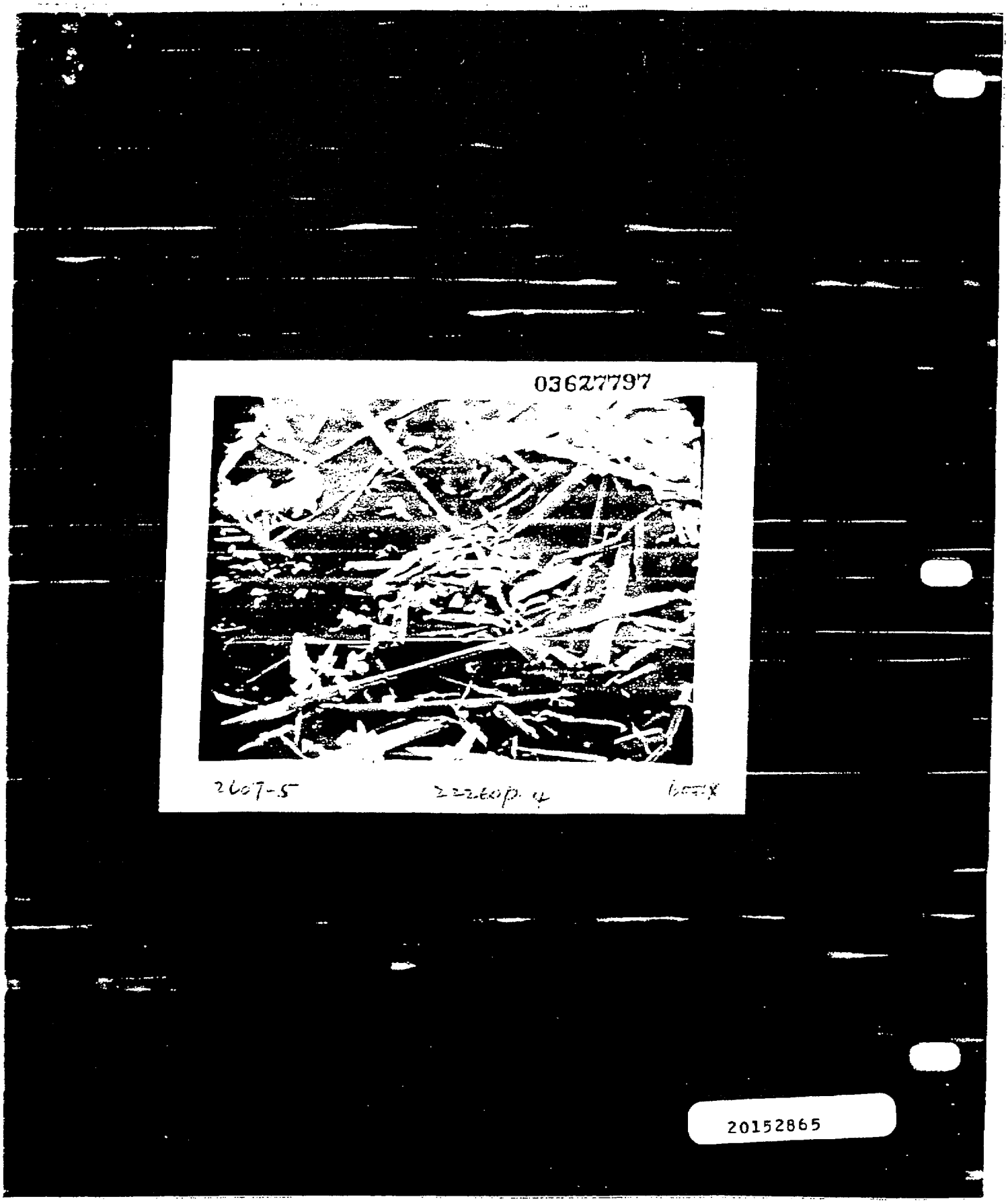
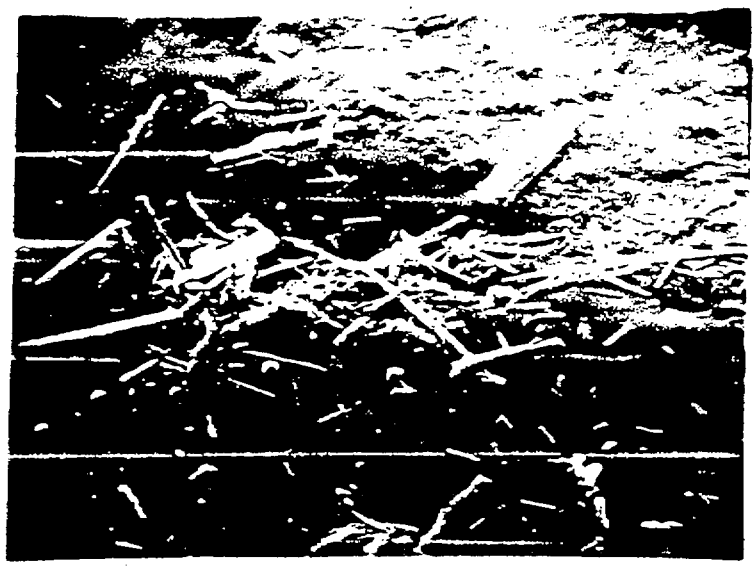

03627797
2607-5
20152865

FIGURE 10

SEM OF RESPIRABLE SIZE TREMOLITE FIBER
PREPARED AND SUBMITTED TO DR. W. SMITH
(22260P-5)

03627798

2606-1 22260P-5 2000X



2606-3 22260P-5 2400X

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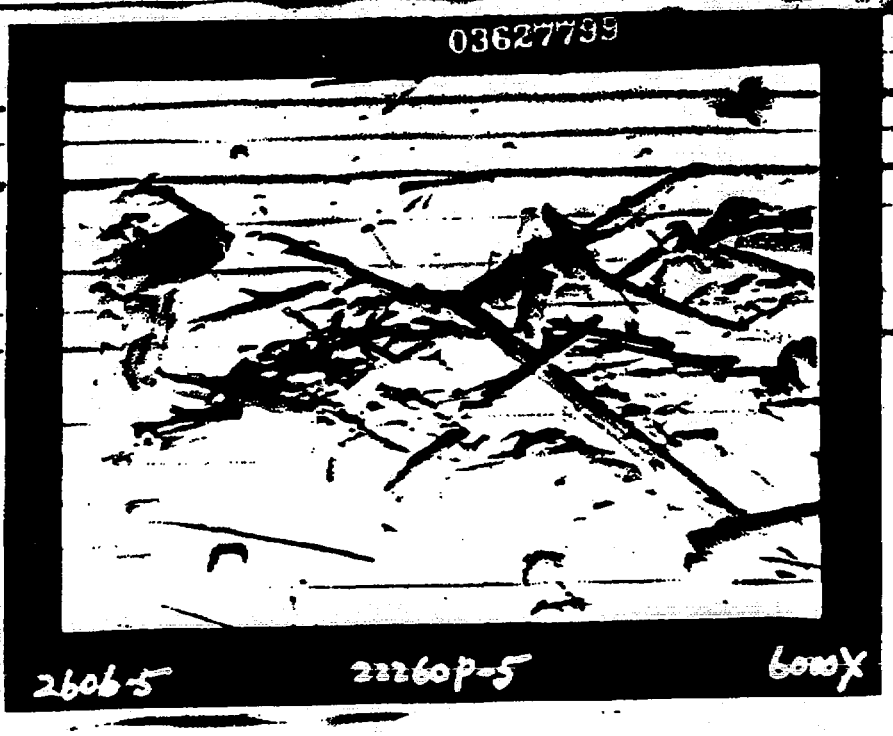


20152867

4863 69

ADMINISTRATIVE

CAMBRIDGE

03627763

TO: J. W. Wolter

DATE: January 6, 1977

FROM: Julie C. Yang

SUBJECT: Tremolite Content in Libby Vermiculite Composites

CC: E. S. Wood
R. L. Oliverio/Libby
H. C. Duecker
F. W. Eaton
File: 71-048

Recently we have determined the tremolite content in Libby #2 composite for the electrostatic spray studies, and found tremolite was in the range around 2.5% which showed a remarkable decrease over the #2 composite we had a year ago. The sample obtained in December 1975 showed about 5% tremolite (report on Libby Ore Evaluation 2/23/76).

If you would like to have the tremolite fiber content of composites of all sizes checked occasionally, we would be glad to do it. The cost of fiber determination for size 1 and 2 is about $80.00 each, and for size 3, 4 and 5 is around $120 per sample.

Julie C. Yang

JCY:mlr

20152831

486492

ORIGINAL ADMINISTRATIVE RECORD

CONSTRUCTION
PRODUCTS
DIVISION

REPORT: 69548
GROUP: Zonolite, BPD
DATE: 3/12/79
CHARGE NO.: 71-194
REQUESTOR: F. W. Eaton
MARKETING OR MANUFACTURING APPROVALS:
NAME: H. A. Eschenbach
APPROVED: H. A. Eschenbach

REQUEST FOR TECHNICAL SERVICE

PROBLEM TITLE: Environmental Evaluation - Air - Fibrous Materials and Tremolite Content

SIGNIFICANCE: The evaluation of workplace air on a periodic basis is necessary to comply with the air sampling section of the OSHA Asbestos Standard.

SPECIFIC OBJECTIVE: Determine fiber counts for filter media and tremolite of the expended vermiculite associated with the air samples.

SUGGESTED APPROACH: Phase contrast microscopy and x-rays.

DEADLINE (Last day information will be of value): 2 weeks.

DETAILS OF PROBLEM: Roof Deck Job Site
Please evaluate Personnel samples from Lone Star Elementary School/New Braunfels, TX:

LS-1 through LS-30

Products and ores involved: Concrete Aggregate - Libby #4

Engineering samples from the same above:

LSE-1 and LSE-2

Products and ores involved: Concrete Aggregate - Libby #4

15171948

ACCEPTED BY RESEARCH DEPT.: [illegible] DATE: 3/13/79
ASSIGNED TO: [illegible]
ADDITIONAL NOTES: Original to Library - H. C. Duecker, H. A. Eschenbach, F. W. Eaton, J. W. Wolter, and B. R. Williams

131Z02172

REQUEST FOR TECHNICAL SERVICE

FOR DISCUSSION

T&A 69548
GROUP ZONOLITE BPD
COST $750
DATE 3/16/79

SUMMARY:

Thirty-two air samples from Lone Star Elementary School in New Braunfels, Texas, were evaluated for their respirable asbestos content, and the results are attached.

The expanded vermiculite used in this study was analyzed for the rock and tremolite content. The results are:

Rock Content	
2.7 >d >1.0	1.06%
d >2.7	0.27%
Total	1.33%
Asbestos Fiber Content	0.0092%

Julie C. Yang

JCY:mlr

15171949

131Z02173

131Z02174

GRACE

AIR SAMPLING RECORD SHEET

(1)

HEALTH, SAFETY & TOXICOLOGY DEPARTMENT

PLANT LOCATION Lone Star Elem. School / New Braunfels, Tx

CONTAMINANT Fiber

SAMPLING BY F. W. Eaton

DATE: 3-6-79

SAMPLING CONDITIONS OUTSIDE Clear 70s

INSIDE DRAFT

VISIBLE DUST

HOUSEKEEPING

TYPE OF SAMPLING: PERSONNEL ✓ ENGINEERING

MEMBRANE (Size & Type)

IMPINGER (Soln & Vol)

PUMP (Type & Model)

Sample Number	Employee Name	SS Number	Job Location & Description	Pump Number	Pump Off	Pump On	Sampling Time	Flow Rate	Total Sampled Volume	Lab Evaluat
										Fibers/cc
LS-1	Art		Emptying Conc. Ag L-4 Bag & Cement Bags into Charging Hopper	RAC I	1043	1030	13	1.6		0.16
LS-2	Art			RAC II	1043	1030	13			1.99
LS-3	Homer		Tieing Empty Cement & Conc. Ag. Bags Handling Conc. Ag. Bags in Trailer	7	1047	1032	15			1.14
LS-4	Tony		Mixer Operator	8	1120	1037	43			0.40
LS-5	Miguel		Hose Man on Roof		1207	1050	77			0.17
LS-6	Same	As LS-1			1057	1043	14			1.22
LS-7	"	" LS-2			1057	1043	14			2.14
LS-8	"	" LS-3	Note 11 Filter Fell off		1109	1047	22			0.39
LS-9	"	" LS-1			1107	1057	10			2.14
LS-10	"	" LS-2			1107	1057	10			2.57
LS-11	"	" LS-1			1125	1107	18			0.47
LS-12	"	" LS-2			1125	1107	18	✓		1.43

Additional Comments: (1) San Antonio L-4 Lot No. SNB59D
" " L-4 " " SNC19E
SNB59E
SNC39E
SNC19D
Conc. Ag. L-4 in GCF Bags.

Laboratory Evaluation by: Jean P. Wallace

Date: 3/15/79

GN 172109
Shipped 1-24-79
Yield 56.4 g/T
Rock 20.1%

Additional Comments on Sheet 2 of 3

15171950

GRACE

AIR SAMPLING RECORD SHEET

HEALTH, SAFETY & TOXICOLOGY DEPARTMENT

PLANT LOCATION Lone Star Elem School

CONTAMINANT Fiber

SAMPLING BY F. W. Enton

DATE: 3-6-79

SAMPLING CONDITIONS:

OUTSIDE _____

INSIDE DRAFT _____

VISIBLE DUST _____

HOUSEKEEPING _____

TYPE OF SAMPLING:

PERSONNEL ✓ ENGINEERING _____

MEMBRANE (Size & Type) _____

IMPINGER (Soln & Vol) _____

PUMP (Type & Model) _____

Sample Number	Employee Name	SS Number	Job Location & Description	Pump Number	Pump Off	Pump On	Sampling Time	Flow Rate	Total Sampled Volume	Lab Evalua
S-13	SAME AS LS-3				1131	1113	18	1.6		Fiber/ 1.19
S-14	" "	LS-4			1157	1120	37			* See comment on back page
S-15	" "	LS-1			1139	1125	14			2.14
S-16	" "	LS-2			1139	1125	14			*
S-17	" "	LS-3			1153	1131	22			*
LS-18	" "	LS-1	NOTE: FILTER FELL OFF		1150	1139	11			4.28
S-19	" "	LS-2			1150	1139	11			3.89
LS-20	" "	LS-1			1201	1150	11			5.05
LS-21	" "	LS-2			1201	1150	11			5.44
LS-22	HOMER		HANDLING BAG 1-4 IN TRAILER FROM SIDE DOOR & TIEING UP EMTY BAGS.	RAC I	1338	1308	30			*
LS-23	HOMER			RAC II	1338	1308	30			1.14
S-24	SAME AS	LS-4		1455	[illegible]	312	103	✓		0.06

Additional Comments (2) APPLICATOR — TURNER ROOFING & SUPPLY Co. SAN ANTONIO

(3) THIS JOB REQ'D 520 - 6CF BAGS OF 1-4

(1) MIXER VENT DISCH. PARTICULATES UP AT ROOF LEVEL

(3) TEXCO SOLD TRAILER TO TURNER. CPD SHOULD PAY TO HAVE "VERMICULITE" AND "HEAVY COM. [illegible] ANOTHER LOAD OF VERMICULITE" PAINTED OUT. IT LOOK LIKE "HEAVY" NOT A GOOD GUESS AND

Laboratory Evaluation by: Jean P. Wallace

Date: 3/15/79

131Z02175

15171951

GRACE

AIR SAMPLING RECORD SHEET

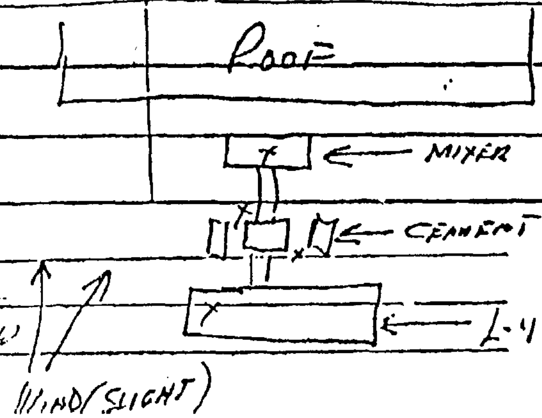
HEALTH, SAFETY & TOXICOLOGY DEPARTMENT

PLANT LOCATION: Lone Star Elem. School
CONTAMINANT: Fiber
SAMPLING BY: F. W. Eaton
DATE: 3-5-79

SAMPLING CONDITIONS:
OUTSIDE ______
INSIDE DRAFT ______
VISIBLE DUST ______
HOUSEKEEPING ______

TYPE OF SAMPLING:
PERSONNEL ✓ ENGINEERING ✓ (2)
MEMBRANE (Size & Type) ______
IMPINGER (Soln & Vol) ______
PUMP (Type & Model) ______

Sample Number	Employee Name	SS Number	Job Location & Description	Pump Number	Pump Off	Pump On	Sampling Time	Flow Rate	Total Sampled Volume	Lab Evaluation (Fiber/cc)
LS-25	Same As LS-5				1515	1323	112	1.6		0.11
LS-26	" "	LS-23			1351	1338	13	↓		2.30
LS-27	" "	LS-24			1351	1338	13			1.97
LS-28	" "	LS-23			1411	1351	20			1.50
LS-29	" "	LS-24			1411	1351	20			0.86
LS-30	F. Eaton		Walking Around Construction Area, Tending Sampling Pump & Keeping Out of Dusty Areas	RAC I	1530	1417	73	✓		<0.03
LSE-1	Engineering Sample		Sample Located Adjacent to Playground 150'± Upwind of Mixer	6	1458	1148	130	1.6		0.16
LSE-2	Engineering Sample		Sample Located Mid Way in Vermiculite Trailer	5	1441	1330	71	1.6		0.4[illegible]



L-4 Trailer

Additional Comments:
*Unable to read because of over-fogging * JPW

Laboratory Evaluation by: Jean P. Wallace
Date: 3/15/79

(6) Man emptying bags of cement & L- wore two (2) pumps. Filter over right shoulder showed heavier dust loading

131Z02176

15171952

(14)

NEW BRAUNFELS MIDDLE SCHOOL - NEW BRAUNFELS TX 3-6-79

① ART — EMPTYING CEMENT & L-4 BAGS INTO CHARGING HOPPER

14	X	1.53	=	21.42
14	X	1.83	=	25.62
17	X	0.75	=	12.75
17	X	2.51	=	42.67
13	X	2.30	=	29.9
13	X	1.64	=	21.32
15	X	3.42	=	51.30
12	X	3.56	=	42.72
18	X	1.66	=	29.88
18	X	2.37	=	42.66
151				320.24

$$TWA = \frac{320.24}{151} \times \frac{7}{8} = 1.856 \text{ f/cc}$$

② TOMY — MIXER OPERATOR

40	X	0.53	=	21.2
40	X	0.32	=	12.8
80				34

$$TWA = \frac{34}{80} \times \frac{7}{8} = 0.372 \text{ f/cc}$$

① 15171953

131Z02177

(3) HOPPER — MOVING L-4 BAGS FROM OPEN TRAILER TO HOPPER & TIEING UP EMPTY CEMENT L-4 BAGS

18 x 0.24 =

TEST NOT VALID

(4) MIGUEL — HOSE MAN ON ROOF

L0.03 f/cc

(ONLY ONE SAMPLE)

15171954

131Z02178

CAMBRIDGE 12/17/79

TO: H. C. Duecker

DATE: November 26, 1979

FROM: Julie C. Yang

SUBJECT: Improvement in Fiber Release with Consumer Products - Test for Uniform Application of Binder

CC: F. W. Eaton
O. M. Favorito
W. R. Hanlon
D. Raab
R. E. Schneider
R. M. Vining
D. D. Walczyk
C. T. Walloch
B. R. Williams
J. W. Wolter
E. S. Wood
File: 70-711

Attic Fill AI with the silver-doped binder, both hand and plant sprayed were analyzed for the uniformity of binder distribution. The air-fiber counts of the simulated attic test results are attached for comparison.

It is demonstrated that the thorough spraying condition (such as hand-spray in this case) can reduce the air-fiber counts to <0.1 f/ml, 8 hours TWA.

The distribution of the silver ions showed that the hand-sprayed material was fairly even in a narrow range with a low standard deviation whereas the plant-sprayed material had a broad distribution with a significant portion with no coating at all.

We have completed the objective A outlined in H. C. Duecker's memo to E. S. Wood, 9/6/79. It is recommended to proceed with Objective B, modifying the spraying delivery and other conditions by the Process Engineering group using the silver-doped binder. Discussions with A. Stockett will be carried out to establish a criteria for the best utilization of our analytical data.

MATERIAL

I) Unbound AI (L-1)

II) Bound AI - Using present plant spraying procedure (0.2 qt/CF & 0.5 qt./CF)

III) Bound AI - Using best hand-spraying (0.2 qt/CF & 0.5 qt/CF)

15103600

108Z01201

EXPERIMENTAL

The CMC binder was doped with a trace of silver nitrate, then the silver ions were extracted from the vermiculite samples after application with hot 1% nitric acid using a procedure developed recently in the laboratory (J.C.Yang to H.C.Duecker, 10/18/79).

The Ag^+ concentration was analyzed by ICAP method (Inductively Coupled Argon Plasma) spectroscopy at Jarrell-Ash, Waltham, Massachusetts. Data is presented in Figure 1, the statistical analysis chart. The actual analytical results are also attached.

The air-fiber counts of these samples in simulated attic test at Weedsport are summarized in Table 1.

STATISTICAL ANALYSIS

Vermiculite Bag Number	Extracted Sample No.	Mean ($\bar{X} \times 10^3$)	Std. Deviation ($\sigma \times 10^3$)	% Std. Dev'n. of Samples $\frac{\sigma}{\bar{X}}$	Item (N)
1) Hand-sprayed (0.2 qt/CF)					
1	1 - 6	38.0	5.76	0.152	6
2	7 - 12	42.7	10.6	0.248	6
3	13 - 42	33.6	11.5	0.343	30
4	43 - 48	32.8	9.68	0.295	6
5*	49 - 54	10.2*	7.78*	0.765*	6*
Ave. (excluding group 5) of the whole series		35.2	10.9	0.309	
		--	--	--	--
	Total:				48

*Calculated t-value for #5 vs. Total (excluding #5) is 7.06. The tabulated t-value for N = 48 (99.9% confidence limits) is 3.50. Thus #5 is statistically and significantly different than the rest of the group. It was thus dropped from all the remaining calculations.

15103601

Vermiculite Bag Number	Extracted Sample No.	Mean ($\bar{X} \times 10^3$)	Std. Deviation ($\sigma \times 10^3$)	% Std. Dev'n. of Samples $\bar{X}$	Item (N)
2) Hand-sprayed (0.5 qt/CF)					
6	55 - 60	61.2	10.9	.179	6
7	61 - 66	64.8	10.9	.168	6
8	67 - 96	70.1	10.5	.149	30
9	97-102	62.0	18.6	.301	6
10	103-108	72.8	20.6	.283	6
Ave. of the whole series		67.9	13.1	.192	--
	Total:				54
3) Plant-sprayed (0.2 qt/CF)					
11	201-230	47.6	52.2	1.09	30
12	231-236	35.8	40.4	1.13	6
13	237-242	33.7	40.6	1.20	6
Ave. of the whole series		43.9	48.6	1.10	--
	Total:				42
4) Plant-sprayed (0.5 qt/CF)					
14	243-272	56.7	38.1	0.636	30
15	273-278	87.8	48.5	0.92	6
16	279-284	37.2	28.2	0.758	6
Ave. of the total series		58.4	40.1	0.686	--
	Total				42

OBSERVATIONS and COMMENTS

1. The hand-sprayed sample has $Ag^{\pm}$ concentration (i.e., the binder material) uniformly distributed throughout the whole assemblage with a narrow range of distribution, and a low standard deviation.

2. The bound sample using the present plant spraying procedure showed a very wide distribution of the binders, and a standard deviation 3-4 times of those hand-sprayed material. A significant portion of the plant-sprayed samples at 0.2 qt/CF (11 out of 42) had no binder at all.

3. The air-fiber count results also indicated the material with uniformly distributed binder (in this case, hand-sprayed) gave lower fiber counts than those of plant-sprayed material. If the binder was applied at 0.5 qt/CF level with good distribution, the 8 hour TWA can be reduced to < 0.1 f/ml.

TO: H.C.Duecker
FROM: J.C.Yang

November 26, 1979

CONCLUSION and RECOMMENDATION

1. All the results indicated that a uniformly coated binder, around 0.5 qt/CF, on attic insulation can meet "zero" or <0.1 f/ml fiber count criteria.

2. Objective A in your memo to E. S. Wood, 9/6/79, has been completed.

3. It is recommended to proceed immediately to Objective B, evaluating the spraying variables as outlined in B1, using silver doped binder. We'll follow up the tasks as outlined.

Julie C. Yang/mlr

Julie C. Yang

JCY:mlr
attachments

TO: H. C. Duecker
FROM: J. C. Yang

November 26, 1979

TABLE 1

SUMMARY OF AIR-FIBER COUNT RESULTS

(based on 50 fields / sample except as noted)

Sample Description	Fiber Concentration f/ml (Number of Fibers Found)		
1) Unbound AI	1.0 (4)	0.75 (3)	0.75 (3)
	1.06 (4)	0.53 (2)	0.53 (2)
	0.14 (1)	0.28 (1)	0.14 (1)*
Ave.		0.58	
2) Bound AI, Plant-sprayed 0.2 qt/CF	0.53 (2)	0.53 (2)	0.53 (2)
	0.28 (1)	0.28 (1)	0.56 (2)
	0.56 (2)	0.28 (1)	0.14 (1)*
Ave.		0.43	
3) Bound AI, Hand-sprayed 0.2 qt/CF	0.27 (1)	0.27 (1)	0.27 (1)
Ave.		0.27	
4) Bound AI, Plant-sprayed 0.5 qt/CF	0.56 (2)	0.28 (1)	0.28 (1)
	0.28 (1)	0.28 (1)	0.28 (1)
	0.25 (1)	0.25 (1)	0.25 (1)
Ave.		0.30	
5) Bound AI, Hand-sprayed 0.5 qt/CF	0.14 (1)*	<0.07 (0)**	<0.07 (0)**
	<0.07 (0)**		
Ave.		<0.09	

*Counted for 100 fields

**Counted for 200 fields

JCY:mlr

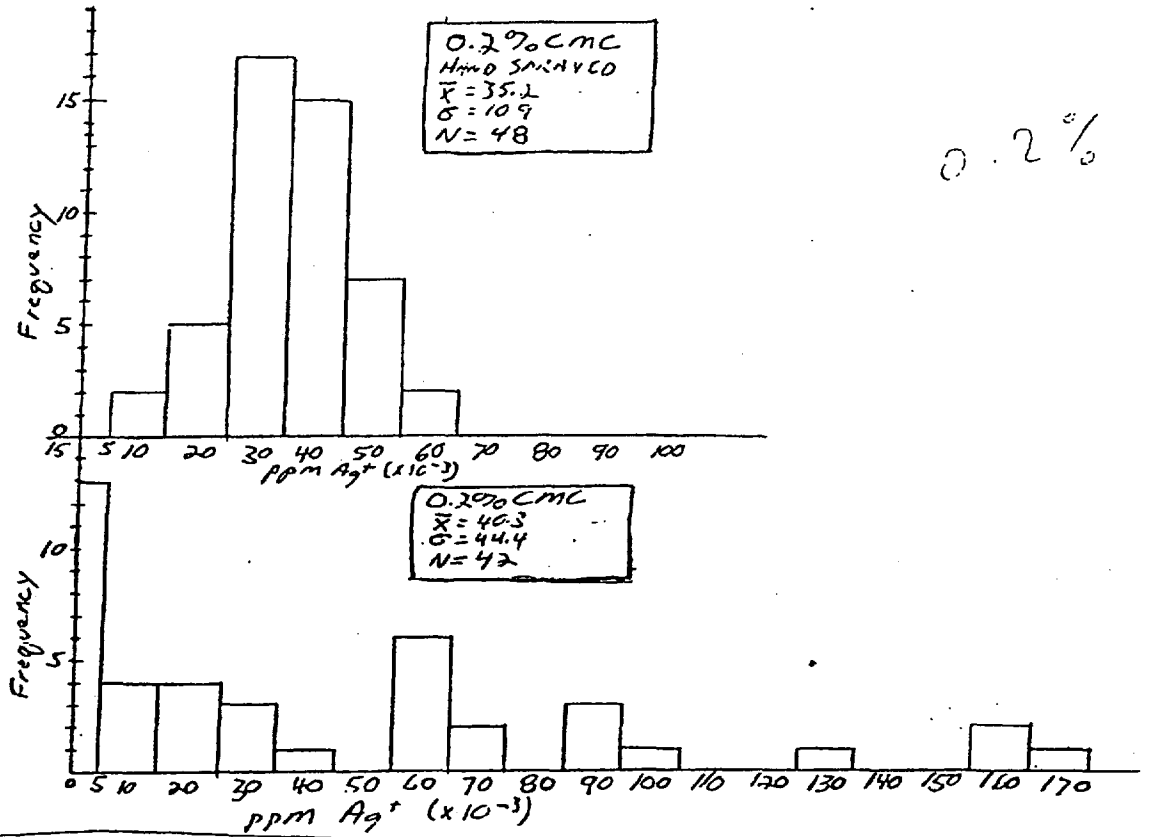
15103604

108Z01205

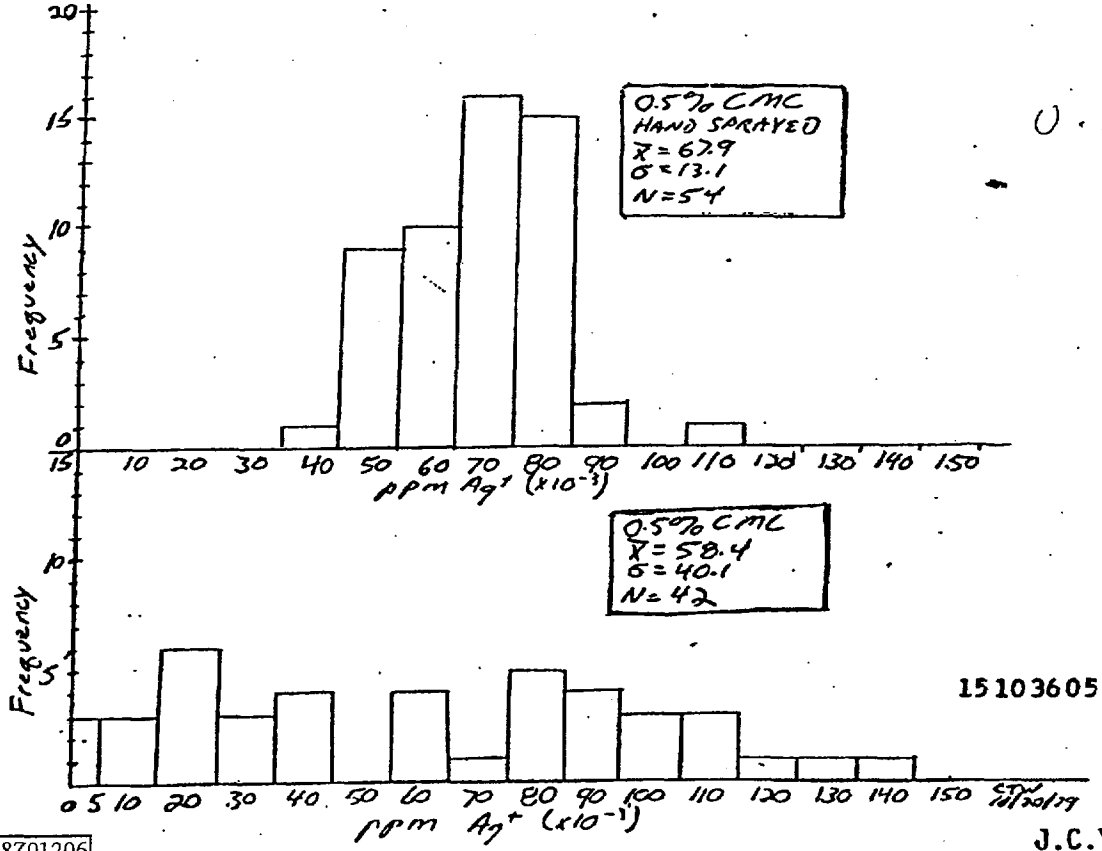
FIGURE 1

03631450

Silver Ion Distributions in Attic Insulation Samples



0.2% CMC



0.5% CMC

15103605

108Z01206

J.C.Yang 11/26/7

W. R. Grace

10/31/79

Order No. 4073

03631451

108Z01207

Sample#	ppmAg	Sample#	ppmAg	Sample#	ppmAg	Sample#	ppmAg	Sample#	ppmAg
1	.040	26	.042	51	.015	76	.072	101	.064
2	.036	27	.032	52	.010	77	.084	102	.050
3	.041	28	.031	53	< .003	78	.065	103	.11
4	.036	29	.036	54	.014	79	.065	104	.075
5	.029	30	.062	55	.076	80	.075	105	.068
6	.046	31	.045	56	.062	81	.066	106	.076
7	.034	32	.044	57	.053	82	.068	107	.053
8	.045	33	.033	58	.068	83	.078	108	.055
9	.039	34	.036	59	.045	84	.079	109	.048
10	.038	35	.049	60	.063	85	.071	110	.050
11	.063	36	.047	61	.065	86	.077	111	.12
12	.037	37	.014	62	.054	87	.064	112	.13
13	.016	38	.014	63	.082	88	.047		
14	.036	39	.024	64	.059	89	.064		
15	.031	40	.034	65	.073	90	.048		
16	.027	41	.019	66	.056	91	.080		
17	.032	42	.015	67	.047	92	.076		
18	.053	43	.046	68	.073	93	.067		
19	.034	44	.042	69	.073	94	.084		
20	.026	45	.026	70	.071	95	.080		
21	.033	46	.021	71	.057	96	.068		
22	.031	47	.028	72	.062	97	.042		
23	.041	48	.034	73	.077	98	.050		
24	.033	49	.020	74	.080	99	.074		
25	.037	50	< .003	75	.085	100	.092		

15103606

W. R. Grace & Company

1/8/79
Order 41125

03031452

Sample#	ppmAg		Sample#	ppmAg		Sample#	ppmAg		Sample#	ppmAg
201	.024		226	.097	0.5	251	.073	0.5	276	.13
202	.021		227	.093	① Plant sprayed	252	.11	② Plant sprayed	277	.079
203	.056		228	.004		253	.11		278	.086
204	.091		229	.17		254	.010		279	.004
205	.021		230	.013		255	.026		280	.059
206	.16		231	.056		256	.10	0.5	281	.025
207	.034		232	.073		257	.060	③ Plant sprayed	282	.079
208	< .003	② Plant sprayed	233	< .003		258	.024		283	.041
209	< .003		234	< .003		259	< .003		284	.015
210	< .003		235	< .003		260	.10		285	< .003
211	< .003		236	.086		261	.017		286	< .003
212	.066		237	< .003		262	.026	Blank	287	< .003
213	.060	0.2	238	.10		263	.097		288	< .003
214	.16	③ Plant sprayed	239	< .003		264	.058		289	< .003
215	.007		240	.026		265	.023		290	< .003
216	< .003		241	.063		266	.12	#5 0.2 cold HS	291	.026
217	.041		242	.013		267	.083	#5 0.2 hot HS	292	.013
218	.057		243	.059		268	.038	#9 0.5 cold HS	293	.064
219	.13		244	.021		269	.088	#9 0.5 hot HS	294	.070
220	.023		245	.076		270	.078	#10 0.5 cold HS	295	.057
221	.004		246	.11		271	.039	#10 0.5 hot HS	296	.070
222	.033		247	.040		272	.006			
223	.057		248	.005		273	.003			
224	.007		249	.015		274	.089			
225	< .003		250	.090		275	.14			

(49-54)
(97-102)
(103-108)

0.2 ① Plant sprayed

1510360?

10820208

Arthur D. Little, Inc. ACORN PARK · CAMBRIDGE MASSACHUSETTS 02140 · (617) 864-5770

April 5, 1977

Dr. Julie C. Yang
Manager, Research Technologies
Construction Products Division
W. R. Grace & Co.
62 Whittemore Avenue
Cambridge, Mass. 02140

Dear Julie: C76494

As we discussed during your visit on March 11, 1977, low magnification transmission electron microscope photographs have been obtained from two representative grid pore openings of samples 22281-1 and 22281-2 to permit an estimate of the percentage of mass attributable to fibers, in particular, amphibole fibers. A previous analysis of these samples, reported on January 24, 1977, identified the presence of fibers, most of which were mineral. These results can be summarized as follows:

	Santa Ana 22281-1	Newark 22281-2
Fibers observed	104	54
Percent amphibole	6	4
Percent other mineral (mostly gypsum)	34	35
Percent ambiguous mineral	35	22
Percent amorphous (organic, glass fiber)	26	39

As some of the ambiguous mineral category may be amphibole, it is prudent to estimate a maximum amphibole fiber content of 10 percent. Due to a slightly larger fiber size, the amphibole fiber volume is about 15 percent of the total fiber volume, which corresponds to 1.6×10^{-12} cm^3 per grid pore opening.

To estimate the relative amount of fibrous material present in the samples, low magnification TEM photographs were obtained from two representative pore openings of both samples. These were assembled into

Arthur D Little, Inc

April 5, 1977

-2-

03627770

Dr. Julie C. Yang
W. R. Grace & Co.

montages, which covered entire pore openings. Particle volumes per pore opening were calculated for the two montages prepared for sample 22281-1A (exhibiting the heaviest particle loading) from the projected surface area and an estimated thickness of each particle, as follows:

0.2μm - particles showing electron beam penetration over whole area

0.5μm - particles showing electron beam penetration at edges

1-2μm - electron opaque particles

From these estimates, the ratio of fiber volume to total particle volume was estimated to be 0.04 percent (0.006 percent for amphibole fibers). For the assumption that the densities of all particles are equivalent, these percentages apply on a mass basis, as well.

From this analysis, we conclude that the amphibole fiber content, on a mass basis, corresponds to less than 0.006 percent of the supplied sample, which represented the insoluble residue fraction of a leached Monokote sample. This estimate should be reliable within a factor of two times.

Please contact me if you have any questions.

Very truly yours,

Ed

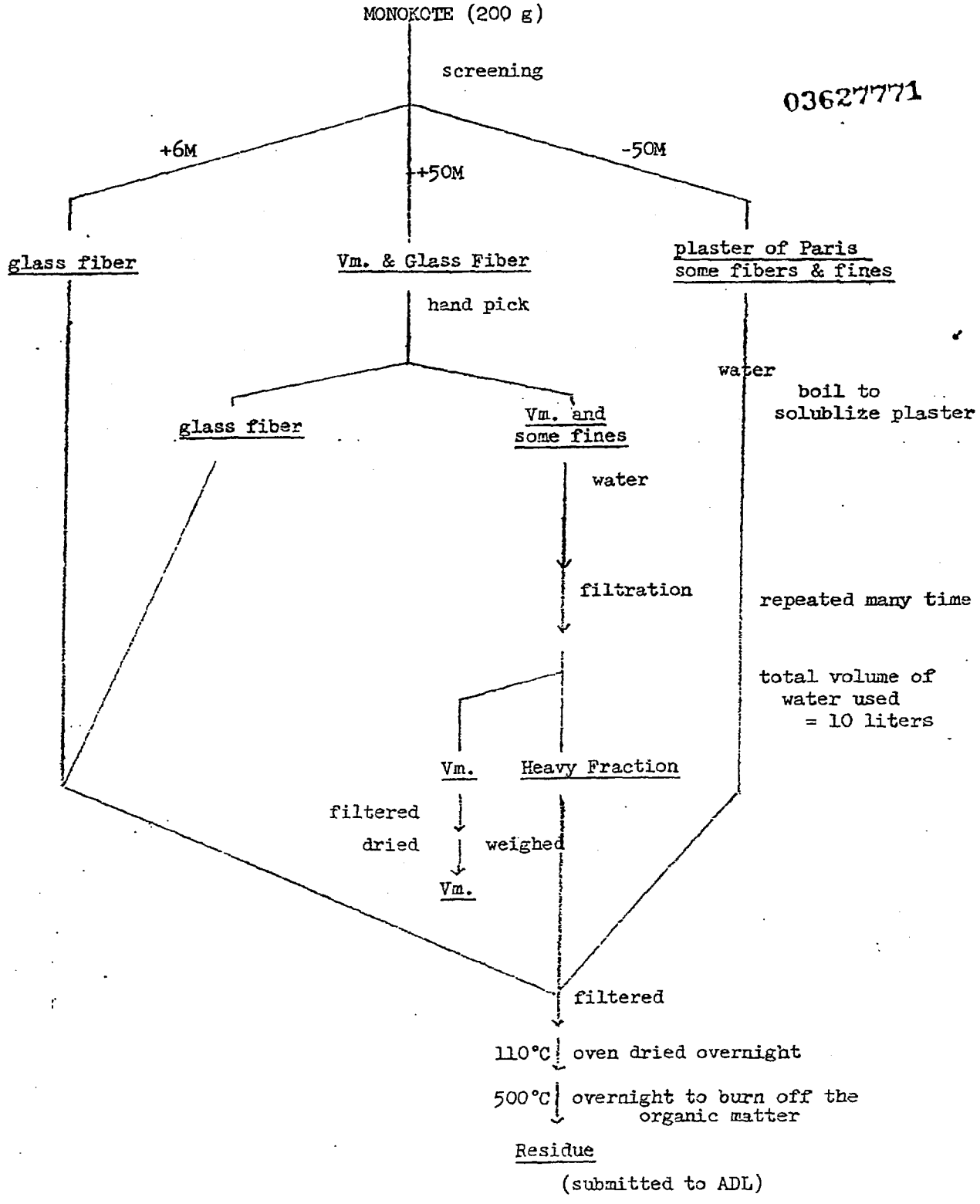
Edward T. Peters

/rdl

20152838

068ETX02094

FIGURE 1 - CONCENTRATION OF FINES IN MONOKOTE®



J.C.Yang:mlr
5/26/77

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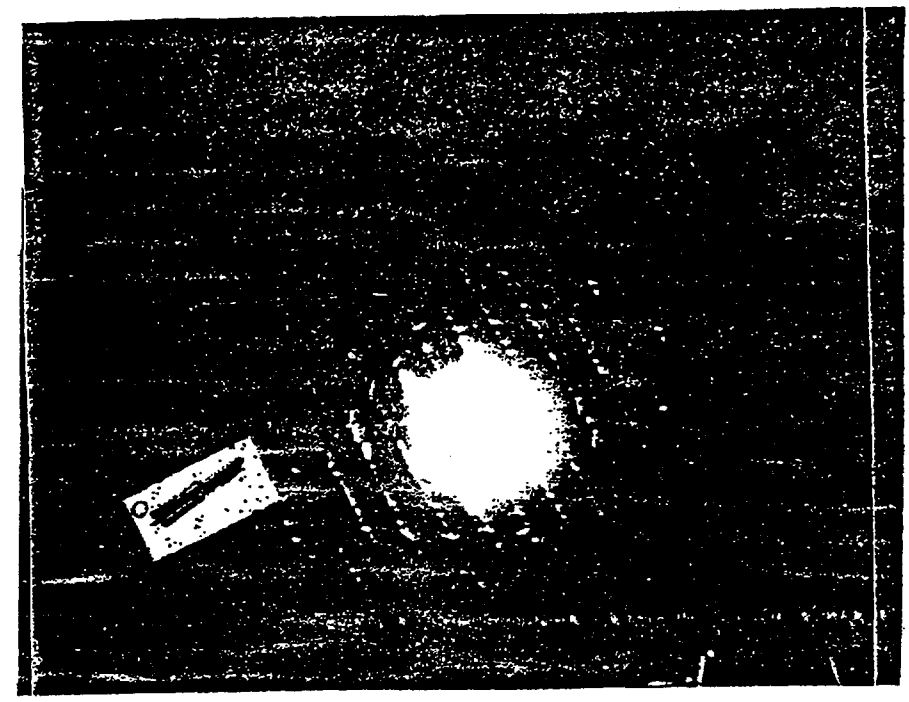

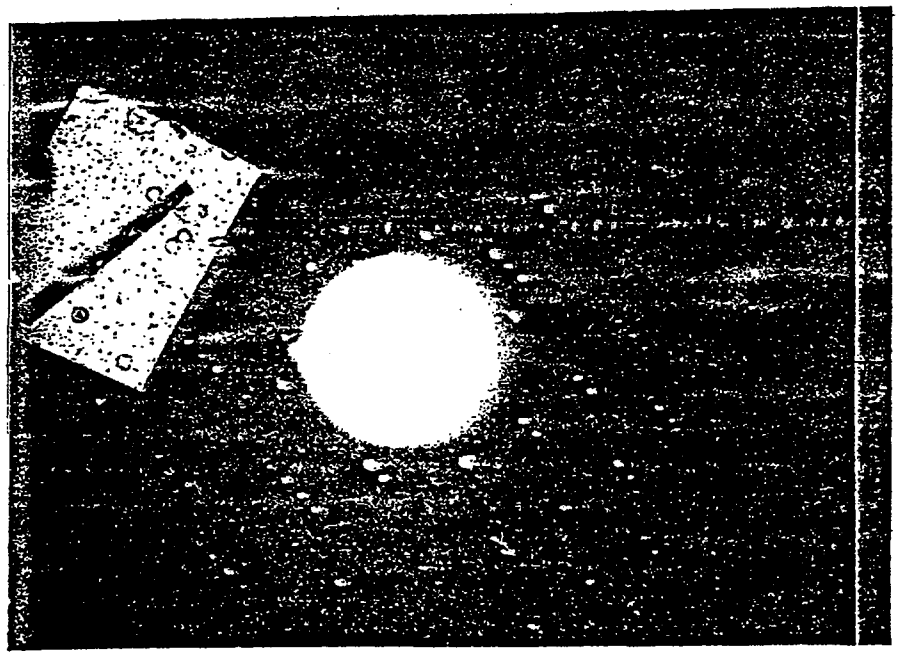

Figure 2. Transmission Electron Image of Fibrous Particles and Corresponding SAED Patterns, Sample 22281-1; 10,000x.

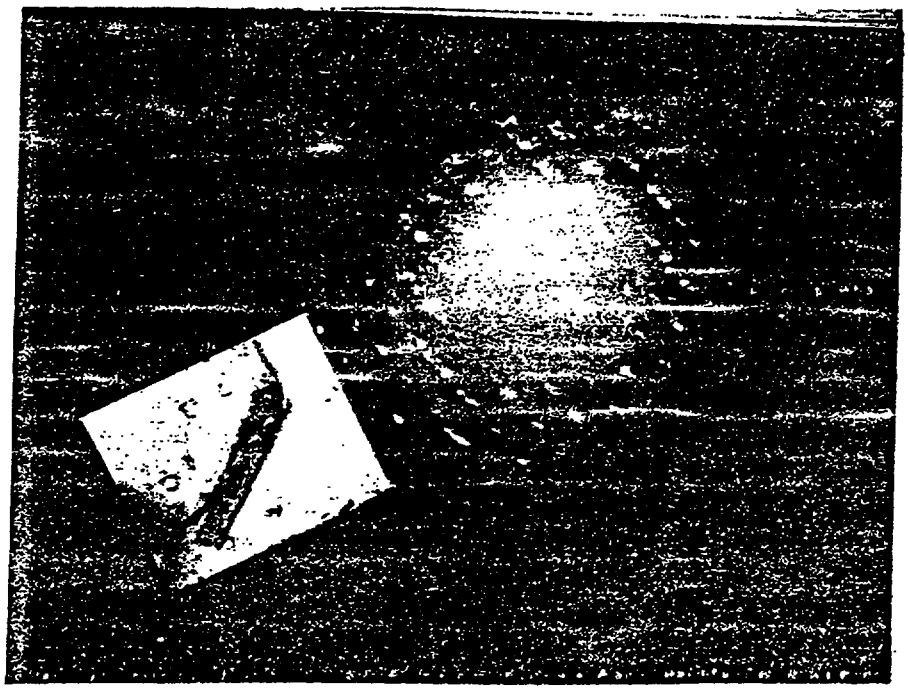

Figure 3. Transmission Electron Image of Fibrous Particles and Corresponding SAED Pattern, Sample 22281-2; 10,000x.

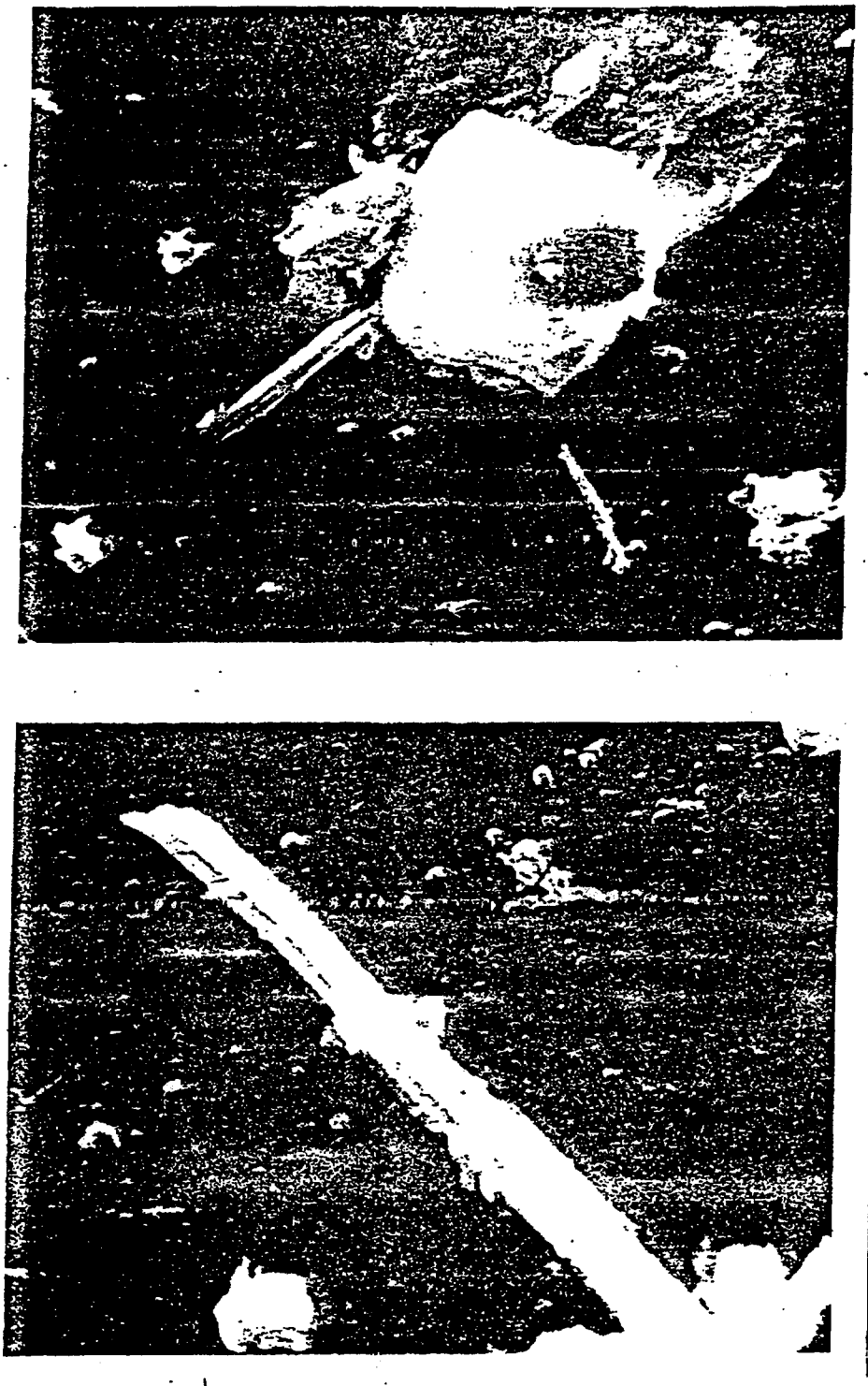

Figure 4. Scanning Electron Micrographs of Fibrous Particles in Sample 22281-1
a) 5500x, b) 5500x

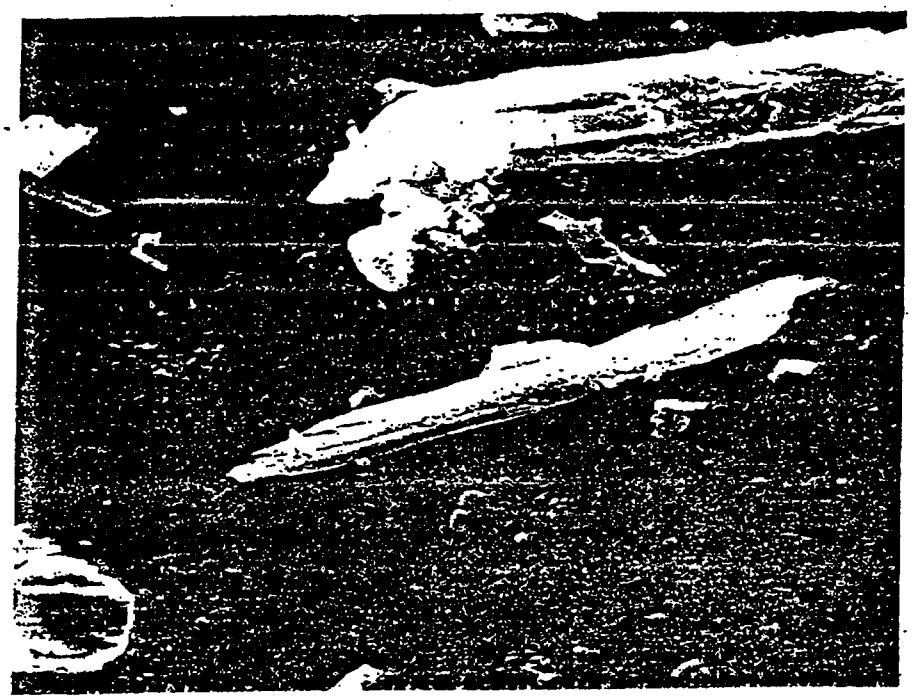

Figure 5 | Scanning Electron Micrograph of a Fibrous Particle in Sample 22281-2, 5500x.

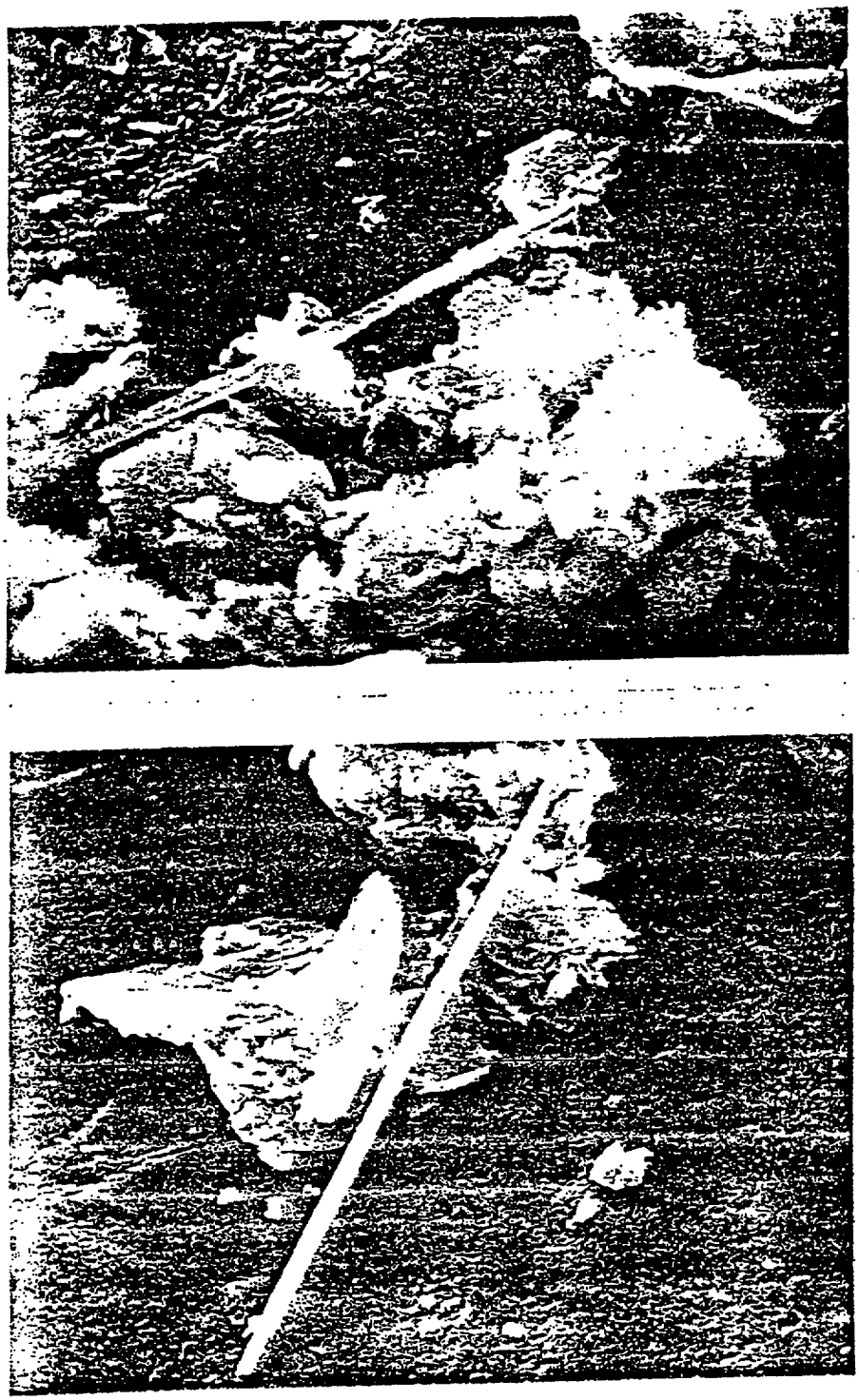

Figure 6) Scanning Electron Micrographs of Fibrous Particles in Sample 22281-1. a) 2400x, b) 1100x

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4-14-77

06020282

CONSTRUCTION PRODUCTS DIVISION

ADMINISTRATIVE RECORD

NUMBER: 50077
GROUP: ZONOLITE-Ore
DATE: February 22, 1977
CHARGE NO.: 71-070
REQUESTOR: J. W. Wolter
MARKETING or MANUFACTURING APPROVAL:
NAME:
APPROVED:

REQUEST FOR TECHNICAL SERVICE

PROBLEM TITLE: LIBBY BAG HOUSE DUST ANALYSIS

SIGNIFICANCE: To identify the composition of dust (mineral make-up), so that attempts can be made in improvements for tremolite fiber removal.

SPECIFIC OBJECTIVE:
To determine percentages of tremolite fiber in the dust samples, and percentage of vermiculite and rock in samples #2 and #4, if possible.

SUGGESTED APPROACH:

DEADLINE (Last day information will be of value):

DETAILS OF PROBLEM:

Four samples are submitted for analysis:

1) Dryer Baghouse Discharge
2) Dryer Cyclone Unders
3) Screen Plant Baghouse (Internal) Discharge
4) No. 4 Product Screen Pan Fraction

ACCEPTED BY RESEARCH DEPT.: [signature] DATE: 2/23/77

ASSIGNED TO: J.P. Walland / S. Vaughan [signature]

ADDITIONAL COPIES: Original to Library, H.C.Duecker, F.W.Eaton, J.W.Wolter, E.S.Wood, R.L.Oliverio/Libby, CPD-T&A, File 71-070

CONFIDENTIAL

15112720

134Z01711

REQUEST FOR TECHNICAL SERVICE

06020283

NUMBER:	50077
GROUP:	ZONOLITE-Ore
ACTUAL COST:	$300.00
REPORTING DATE:	March 3, 1977

SUMMARY:

Four dust samples received from Libby were analyzed for their tremolite content.

Due to the high percentage of vermiculite present in sample 2 (Dryer cyclone unders) and sample 4 (#4 product screen pan fraction), the vermiculite was separated by chemical expansion with 30% H_2O_2 and flotation. The rock portion was then x-rayed for quantitative tremolite determination.

The Baghouse Discharge dusts from Dryer and Screen Plant were found to be extremely fine and to have 15.5% and 14.2% tremolite, respectively, which should be discarded rather than returned back into the concentrate. No attempt was made to determine the other ingredients present.

The Dryer Cyclone Unders showed the presence of 91.5% vermiculite, 7.82% rock and 0.68% tremolite. This material can be returned back into the concentrate directly.

The No. 4 Product Screen Pan fraction showed a composition of 65.4% vermiculite, 31.0% rock and 3.6% tremolite. The recovery of the vermiculite from this fraction may be more difficult on account of the high rock and tremolite fiber content.

CONCLUSIONS and COMMENTS:

See Summary.

EXPERIMENTAL:

1. Separation

About 60 g of sample (#2, #4 only) was expanded with 100 ml of 30% H_2O_2 for two days.

The expanded vermiculite was floated off with water, collected on a filter and dried in an oven at 110°C. overnight.

The rock portion was also dried, weighed, and x-rayed:

Sample I.D. No.	Description	% Vm.	% Rock and Tremolite
22293-2	Dryer Cyclone Under	91.5	8.5
22293-4	#4 Product Screen Pan Fraction	65.4	34.6

2. X-Ray Analysis

All the determinations were made on the measurement of peak intensity at $2\theta = 10.55°$ (d = 8.38Å) in triplicate with background corrections and calibration curve.

Sample No.	Description	% Tremolite
22293-1	Dryer Baghouse Discharge	15.5
22293-2	Dryer Cyclone Under - Rock & Tremolite Portion	8
22293-3	Screen Plant Baghouse Discharge	0.68
22293-4	#4 Product Screen Pan Fraction - Rock and Tremolite Portion	14.2

All samples were collected on January 26, 1977.

15112721

134Z01712

REQUEST FOR TECHNICAL SERVICE

NUMBER:	50077
GROUP:	ZONOLITE-Ore
ACTUAL COST:	$300.00
REPORTING DATE:	March 3, 1977

3. Computation of Mineral Compositions

06020284

Sample No.	Description	Composition	
22293-1	Dryer Baghouse Discharge	Tremolite	5.5%
22293-2	Dryer Cyclone Unders	Vermiculite	91.5%
		Rock	7.82%
		Tremolite	0.68%
22293-3	Screen Plant Baghouse Discharge	Tremolite	14.2%
22293-4	#4 Screen Product Pan Fraction	Vermiculite	65.4%
		Rock	31.0%
		Tremolite	3.6%

Ref: SV - 98172P
JCY - 22294P
X-Ray Patterns % - 42 - a,b,c,d

Julie C. Yang

JCY:mlr

15112722

134Z01713

CAMBRIDGE

487251

TO: E. S. Wood

DATE: April 19, 1977

FROM: Julie C. Yang

SUBJECT: Tremolite Content in ZONOLITE® Products

CC: H. C. Duecker
H. A. Eschenbach
F. W. Eaton
W. R. Hanlon
R. M. Vining
B. R. Williams
J. W. Wolter
C. C. Ou
S. C. Vaughan
File: 71-046

OBJECTIVE

The objective of this study is to determine the tremolite content in all ZONOLITE products made of both Libby and Kearney vermiculites. In a few cases, repetitious analyses were made for product used on job-sites, so that correlation can be made with the fiber counting results.

METHOD

When tremolite is determined from the product as received, in most products tremolite was not found by conventional analytical methods. The trace amount can be determined only when intensive concentration techniques are employed. Tremolite determinations are then made from the fractions by quantitative x-ray diffraction analysis and with the aid of petrographic microscopic examination.

1. Terra-Lite Vermiculites, Verxite, Redi-Earths and Metro-Mixes

The schematic method of analysis and the results have been reported in T&A 50110 with limited distribution. They are also reported here as shown in schemes 1, 2, and 3.

2. Scott Turf Builder

The method of concentration was very similar to that of Terra-Lite Vermiculite scheme #1, except in the water flotation step. A longer soaking period was needed to solubilize all the nutrients present, which was approximately 50% of the total weight.

3. ZIC, Attic Fill, Masonry Fill

Same concentration method as Terra-Lite (scheme #1).

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4. MONOKOTE

Analysis of tremolite in MONOKOTE was the most difficult and time-consuming procedure. The glass fibers were screened off, plaster of Paris was dissolved in water about 50-100 times the weight, expanded vermiculite was floated off, and all the washings were combined, filtered and dried. The filter paper and the organic matter were then burnt off; the remaining residue was x-rayed for the tremolite analysis. Detailed separation and concentration procedure is shown in scheme #4.

5. ZONOLITE 3300

Separation and concentration techniques are similar to that of MONOKOTE, but dilute acid (in HCl) was used to digest the portland cement binder instead of using large excess of water for solubilizing plaster of Paris. The procedure is shown in scheme #5.

RESULTS

A. Tremolite Content in ZONOLITE Products

Kearney

ID No.	Product Description	% Tremolite			
1	ZIC K-4 Kearney	5.466			
2	ZIC K-4/5 B	1.715			
4	Masonry Fill K-4	1.605			
9	Masonry Fill K-3	.0504			
11	MK-4 Kearney 3	<0.08			
13	MK-5 Kearney 3	<0.08			
17	Terra-Lite Kearney	4.319			
18	Terra-Lite T.R.	~~0.016~~ 0.16			
20	Metro Mix 200 T.R.	(as rec'd) 0.398	(dried)* .477		
21	Redi-Earth T.R.	(as rec'd) 0.048	(dried) .071		
23 (5)	Verxite Carrier Grade #4, Kearney (St.Louis)	0.083 (<0.008)			
26	Metro-Mix 300, T.R.	(as rec'd) 0.081	(dried) 0.121		
27	Metro-Mix 350, T.R.	(as rec'd) 0.156	(dried) 0.259		

*Metro-Mixes and Redi-Earths were computed both in as-received basis and oven-dried basis since the product has substantial amount of moisture.

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Libby

ID No.	Product Description	% Tremolite	
10	MK-4 (L-3) West Chicago	< 0.10	
6	Masonry Fill (L4D-18) West Chicago	0.01	
19	Terra-Lite, W. Chicago	0.035	
25	Attic Fill (L-2) W.Chicago	.013	
28	Redi-Earth (L) Santa Ana	(as rec'd) .031	(dried) .051
14	Redi-Earth (L) W. Chicago	<0.02	
15	Metro-Mix 200 (L) W. Chicago	(as rec'd)0.034	(dried)<.043
12	Zonolite 3300 (L-3) W. Chicago	<0.007	
3	Concrete Aggregate (L4D-18) W. Chicago	0.344	
16	Scott Turf Builder (L) Dark	0.009	
22	Scott Turf Builder (L) Light	<0.009	

B. Tremolite Content in Zonolite Job-site Samples

ID No.	Product Description	Location	% Tremolite	
8	ZK Roof Deck (K 4/5 B)	Montgomery, Ala.	2.828	
9	Masonry Fill (K-3)	Columbus, Ohio	0.050	
28	Redi-Earth (L-4)	Forest Service, Santa Ana	0.931	(.051)*
51	Monokote-5 (L-3)	San Diego	<0.106	
54	Masonry Fill (K-4)	W.Palm Beach, Fla.	2.86	
55	ZIC (K-4)	Edison H.S., Miami, Fla.	0.476	
58	Masonry Fill (L-3)	Mashburn & Coe Bldg., Oklahoma	0.250	
57	Monokote-4 (L-3)	Hyatt Regency, Dallas	0 240	

*oven-dried basis

DISCUSSION and COMMENTS

1. Some of the Kearney products showed high "tremolite" content since x-ray diffraction method cannot distinguish massive tremolite (Hornblende?) and fibrous tremolite. Microscopically, most of the Kearney material showed trace or absence of fibers.

2. Tremolite fibers can be reduced if a screened vermiculite is used such as in verxite. We have observed that most of the fibers are concentrated in the fines.

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3. The percentage of tremolite in several samples was expressed in less than a certain value which indicated that tremolite fiber was not detected by our x-ray method. The limit of detection for tremolite by x-ray diffraction technique is about 0.2%. When concentration factors were taken into consideration, the possible maximum tremolite content in each sample was indicated in the analyses.

4. Most of the Monokote showed undetectable tremolite content except #57, an MK-4 product used at Hyatt Regency in Dallas, which showed a 0.24% tremolite; the value has been double checked and is real.

Julie C. Yang

JCY:mlr

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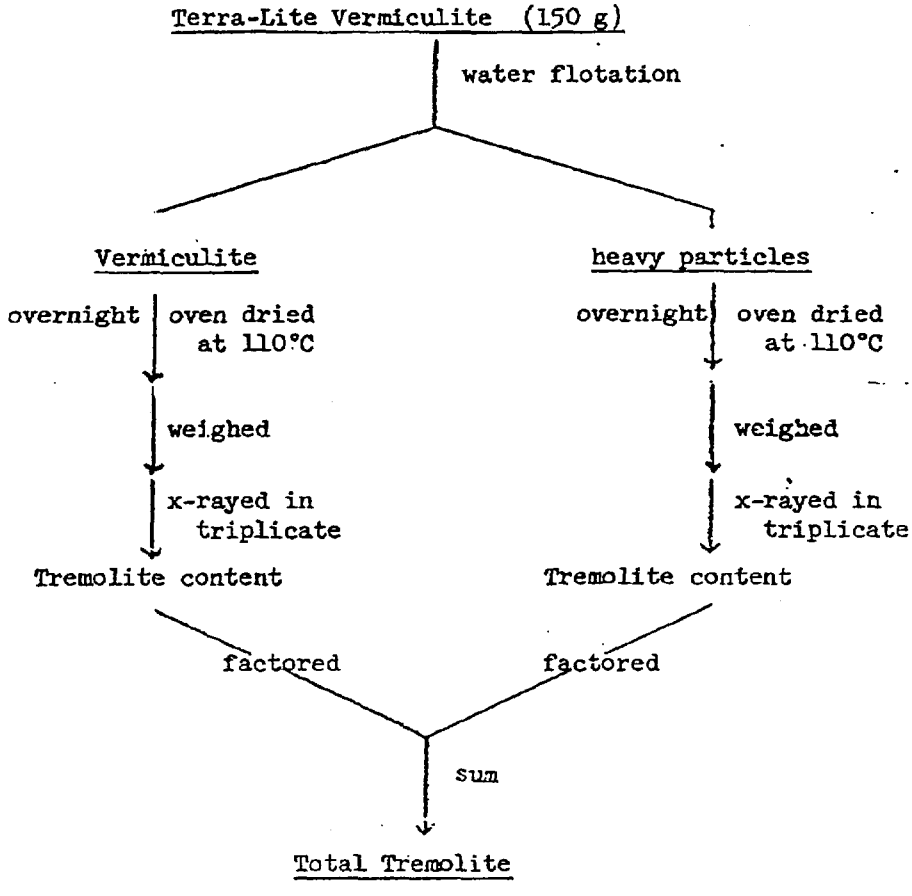
123Z00545

1. SCHEMATIC DIAGRAMS FOR TREMOLITE ANALYSIS

1. Tremolite Determinations in Terra-Lite Vermiculite



Julie C. Yang
April 19, 1977

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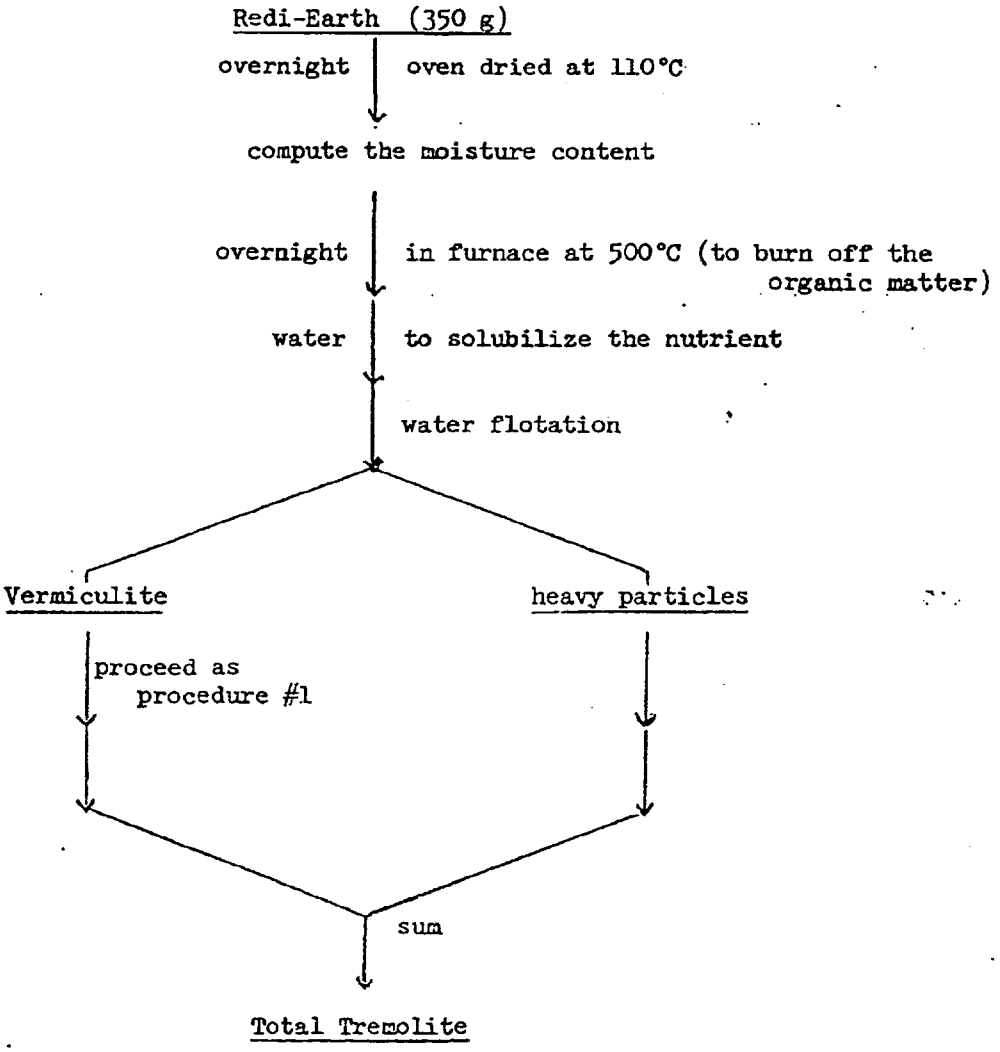
2. Tremolite Determination in Redi-Earth



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Julie C. Yang

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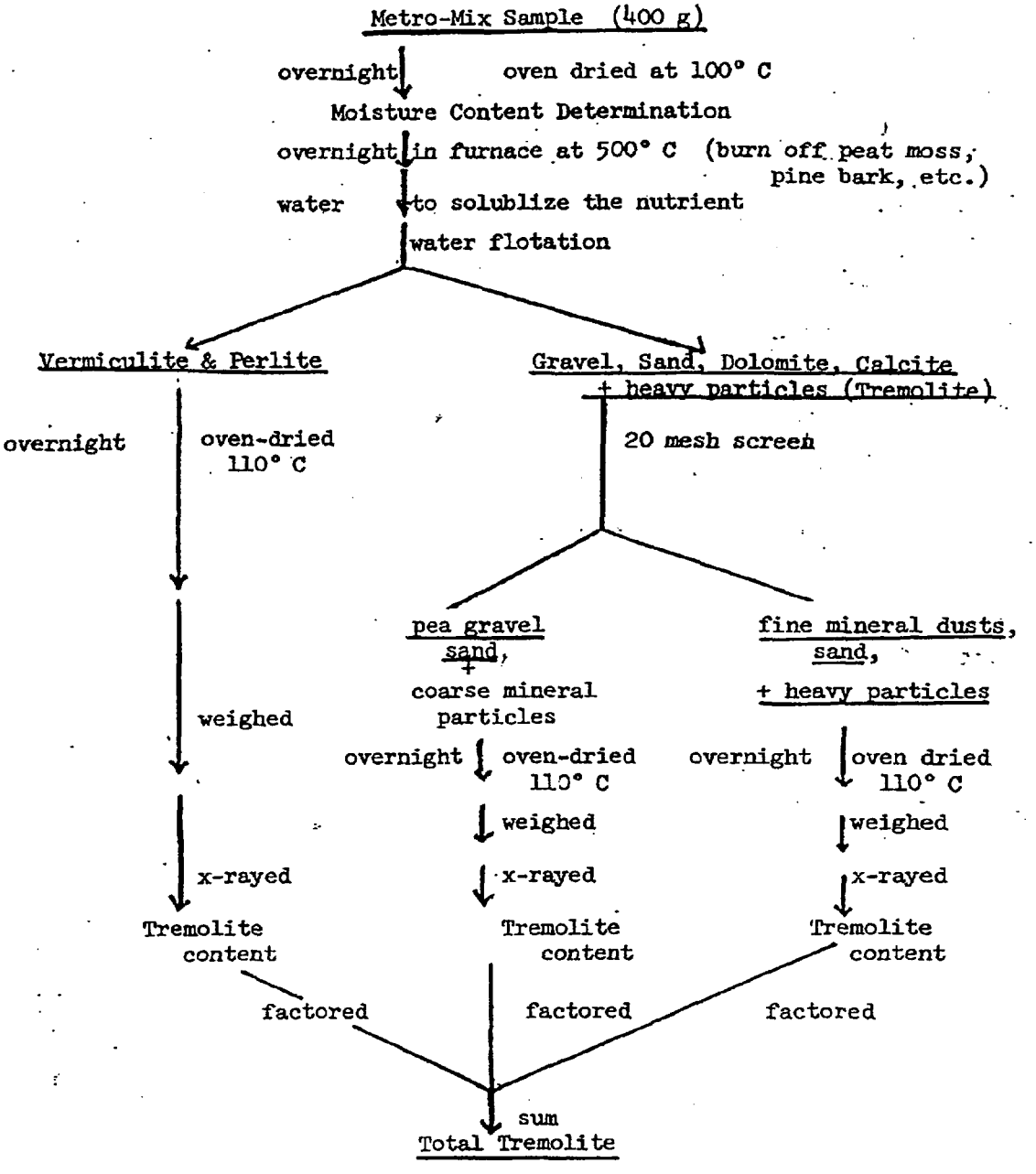
3. Tremolite Determinations in Metro Mix



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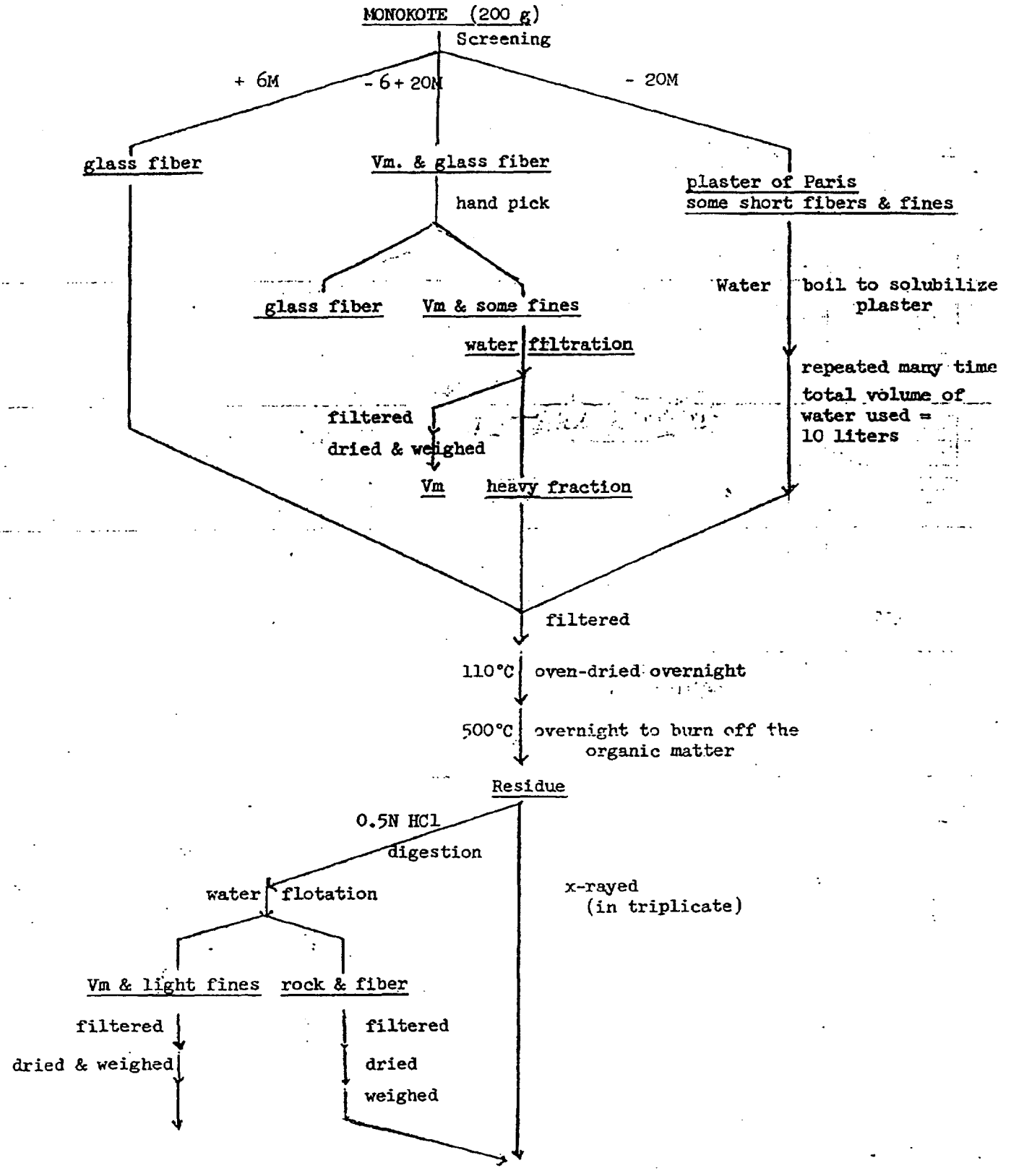
4. TREMOLITE DETERMINATION IN MONOKOTE



Julie C. Yang
April 19, 1977
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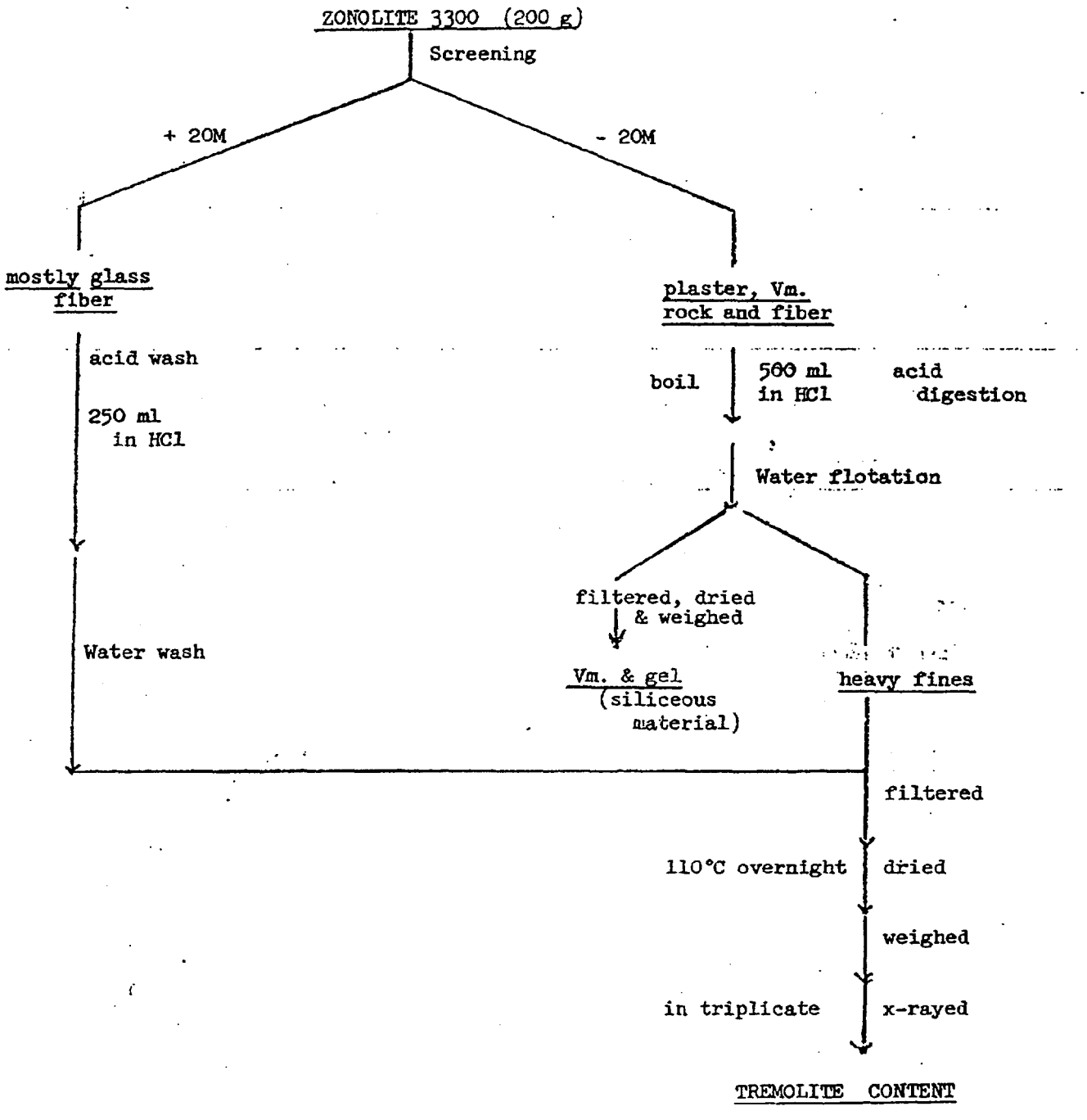
5. TREMOLITE DETERMINATION IN ZONOLITE 3300



Julie C. Yang
April 19, 1977

15152546

123Z00550

CONFIDENTIAL

CAMBRIDGE

TO: E. S. Wood

DATE: May 16, 1977

FROM: J. C. Yang

SUBJECT: Tremolite & Vermiculite Content in Libby & Kearney Ore Deposits and Expanded Vermiculites

CC: R. M. Vining, B. R. Williams, J. W. Wolter, W. R. Hanlon, D. M. Kirven, R. C. Ericson, R. H. Locke, H. C. Duecker, H. A. Eschenbach, B. A. Blessington, C. C. Ou, F. W. Eaton, O. M. Favorito, S. C. Vaughan
File: 71-048/049

Copy to F. Eaton 7/79 (2nd)
A. Crawford 12/79
S. Ahren

OBJECTIVE

The objective of this study is to determine the vermiculite and tremolite content in ore concentrate and expanded vermiculite from the Libby and Kearney mills. A sample of the head feed from the Libby mills, from which all the Libby ore samples were derived, is also analyzed as a check for the effectiveness in fiber removal of the Libby operation.

The samples analyzed below are single samples of concentrate or expanded product, selected at random. We do not know how accurately these samples represent the average with respect to tremolite (or amphibole mineral) content. Further sampling will be required to better establish more typical or average values.

The reported tremolite content may include other amphibole minerals, particularly hornblende, which cannot be distinguished from tremolite.

SAMPLE DESCRIPTION

All the analyses made in this report were single sample analyses. From the materials submitted in 5-10 lb. quantities, they were quartered very carefully and repeatedly until the desired sample sizes (200-300 grams) were obtained, which were expected to be fairly representative. However, the range of variations in field sampling and in the geological formations were not established, so that the results observed may only indicate a ballpark figure with ±10% of accuracy.

To: E.S.Wood
From: J.C.Yang
Date: May 16, 1977

Tremolite & Vermiculite Content in Libby & Kearney Ore Deposits and Expanded Vermiculites

ID No.	Description	Date and Source
99952-31	Ore Concentrate L-1	3/10/77 - R. L. Oliverio
99952-32	Ore Concentrate L-2	
99952-33	Ore Concentrate L-3	
99952-34	Ore Concentrate L-4	
99952-35	Ore Concentrate L-5	3/1/77 - E. D. Lovick
99952-36	Ore Concentrate K-3	3/7/77 - O. F. Stewart
99952-37	Ore Concentrate K-4	
99952-38	Ore Concentrate K-5	
99952-39	Expanded Vermiculite L-1	3/21/77 - F. W. Eaton
99952-40	Expanded Vermiculite L-2	
99952-48	Expanded Vermiculite L-3 (Terra-Lite)	3/9/77 - F. W. Eaton
99952-41	Expanded Vermiculite K-3	3/3/77 - O. F. Stewart
99952-42	Expanded Vermiculite K-4	
99952-43	Expanded Vermiculite K-5	
99952-46	Libby Head Feed - a composite of 3 shifts	3/9/77 - R. L. Oliverio

METHOD

1. Tremolite Analysis of Libby #1 and #2 Concentrate:

   Since the fiber bundles and the rock aggregates are unusually large, tremolite fiber bundles and rocks were first separated by hand-picking of a carefully quartered sample. The vermiculite was then separated from the rock by screening. Rocks and fines in the -50 mesh fraction were x-rayed for quantitative determination of tremolite. The total tremolite was obtained as the sum of factored portions from hand-picked and the fine portions. The scheme of analysis is shown in Figure 1.

2. Tremolite Analysis of #3 Ore Concentrate:

   The concentration of rock fines and tremolite fiber fractions are shown in Figure 2. Vermiculite was separated by chemical exfoliation with 30% H_2O_2, followed by water flotation.

15152532

123Z00536

To: E.S.Wood
From: J.C.Yang
Date: May 16, 1977

Tremolite & Vermiculite Content
in Libby & Kearney Ore Deposits
and Expanded Vermiculites

3. Tremolite Analysis of #4 and #5 Ore Concentrate:

A set of finer screens than the ones used in analyzing #3 ore was selected for this separation. A diagram of the procedure is shown in Figure 3.

4. Tremolite Analysis of Expanded Vermiculite (size #2 to #5)

The expanded vermiculites are easier to work with since they were expanded already and the percentage of rocks and fines were lower than those of the corresponding ore concentrates.

The procedure for analyses is shown in Figures 4 and 5, respectively.

5. Tremolite Analysis of the Head Feed:

The head feed sample from the Libby mill was obtained one day before the ore composite collection from the screening plant, and is the starting material from which the ore composites were obtained. The analysis was more complicated than the others since the size varied over a wide range and the non-vermiculite portion was very high. The tremolite concentration procedure is shown in Figure 6.

6. Vermiculite Analysis of Ore Concentrates:

To cross-check the vermiculite analysis from the scheme shown in Figures 2 and 3, for ore composites #3 and #5, a 100 g ore sample was taken and expanded in a furnace for 5 minutes at 1500°F., then allowed to cool at ambient conditions for half an hour. In general, a weight loss of about 7% resulted from heat expansion. By previous experience, a higher vermiculite yield will result from chemical expansion since some of the poorly weathered vermiculite will not readily respond to heat expansion but will expand in H_2O_2. The complete analyses of the Libby and Kearney vermiculites are shown in Tables 1 and 2.

7. Evaluation of Fine Fiber Content:

In Table 1, a breakdown of the tremolite fiber in ore concentrate by size fraction is also shown. The fines (-50M for size L-1 to L-3; -100M for size L-4 and L-5) can be considered to be the maximum limit of the respirable fiber portion (provided no further vigorous mechanical degradation of the material takes place in handling).

We have also hand-picked the fiber bundles from two L-2 ore concentrate samples and run them through the air-elutriation column built in the laboratory. We then collected the airborne particulate through a series of screens and then on a wet filter under vacuum. The screens used were graded to eliminate the blockage of the filter by large dust aggregates and long fibers. This experiment indicated the fiber bundles were fairly stable. At the end of 30 minutes of the air elutriation, the results are as follows:

To: E.S.Wood
From: J.C.Yang
Date: May 16, 1977

Tremolite & Vermiculite Content in Libby & Kearney Ore Deposits and Expanded Vermiculites

Sample Description	% wt. on 325 mesh screen	% wt. on wet filter	Respirable Dust
Tremolite, hand-picked from SL-2	0.29	0.05	0.34
Tremolite, hand-picked from CL-2	0.21	0.04	0.25
Observation:	mostly fiber some larger than 30 μ	fiber and vermiculite dust	

The fine sized dust content average for the two samples was approximately 0.3%, corresponding to .0076% of the total sample. Thus, it is concluded that the amount of respirable size tremolite fiber present in the L-2 ore composite must be less than 0.01%.

No attempt was made to determine the respirable size fiber content from the Kearney ore since most of the Kearney tremolite is massive and difficult to be distinguished from hornblendes present.

In the expanded product, the tremolite fiber contents found in the Libby vermiculites as shown in Table 2 were primarily fine sized. Very few small bundles were observed which demonstrates the effectiveness of fiber removal by the stoner.

COMMENTS

1. The tremolite content of L-1 and L-2 were reduced to about half the amount of those analyzed in 1976 (see Research Report on Libby Ore Evaluation - J. C. Yang to H. C. Duecker, 2/23/76). The tremolite content of L-3 and L-4 has apparently not improved since early 1976. However, the vermiculite platlets were much cleaner presently with less dust particles adhered to the surface than those of 1976.

2. This is the first time that Kearney ores and expanded vermiculites of all sizes were analyzed for their total tremolite content by the x-ray diffraction method. Unfortunately, the x-ray diffraction patterns of the fibrous and massive forms of tremolite are identical and in fact cannot be distinguished from other amphibole minerals, particularly hornblende. More sophisticated analytical methods using electronmicroscopic techniques and related structural and elemental analysis such as TEM, SEM, SAED and EDAX are needed to pinpoint the exact nature of the amphibole minerals present.

15152534

123Z00538

To: E.S.Wood
From: J.C.Yang
Date: May 16, 1977

Tremolite & Vermiculite Content in Libby & Kearney Ore Deposits and Expanded Vermiculite

3. The fine size (and potentially respirable size) tremolite fiber contents in the Libby ore composites were very low (in the order of 0.01%) for L-2 ore concentrate. Fiber bundles usually remain intact under normal operations and are concentrated in the stoner. Some of the small fibers present between vermiculite plates may be loosened during the expanding operation, the amount yet to be determined. Another possible source of respirable size fibers in expanded product is the breakdown of fiber bundles during heat expansion. This will be investigated shortly. When all the sources are identified and the approximate amounts become known, a method for more effective removal or reduction can be sought with some confidence.

Julie C. Yang

JCY:mlr

attachments

15152535

123Z00539

To: E.S.Wood
From: J.C.Yang
Date: May 16, 1977

Tremolite & Vermiculite Content in Libby & Kearney Ore Deposits and Expanded Vermiculite

TABLE 1

Tremolite Content of Ore Concentrate

ID No.	Description	Date	% Vm.	% Tremolite		% Total Tremolite*
-31	L-1	3/10/77	91.7	(+50M) (-50M)	1.2 .005	1.2
-32	L-2	3/10/77	91.2	(+50M) (-50M)	2.5 .018	2.5
-33	L-3	3/10/77	78.1	(+50M) (-50M)	.653 .013	0.7
-34	L-4	3/1/77	70.1	(+70M) (-70 +100M) (-100M)	1.495 .232 .009	1.7
-35	L-5	3/1/77	63.9	(+70M) (-70 +100M)	.119 1.016 1.913	3.0
-36	K-3	3/1/77	72.0	(+50M) (-50M)	1.60 .158	1.8
-37	K-4	3/1/77	75.1	(+70M) (-70 +100M) (-100M)	8.903 .554 .492	10.0
-38	K-5	3/1/77	76.6	(+70M) (-70 +100M) (-100M)	0.874 2.070 13.034	15.9
-46	Head Feed, Libby	3/9/77	7.0**	(+6M) (-6 +20M) (-20 +70M) (-70M)	1.302 .684 1.235 .609	3.8

*Includes all amphibole minerals.

**The material floated after expanded with 30% H_2O_2.

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CONFIDENTIAL

123Z00540

To: E.S.Wood
From: J.C.Yang
Date: May 16, 1977

Tremolite & Vermiculite Content in Libby & Kearney Ore Deposits and Expanded Vermiculite

TABLE 2

Tremolite Content of Expanded Vermiculite

ID No.	Description	Date Collected	% Vermiculite	% Tremolite
99952-39	L-1	3/18/77	97.7	0.074
99952-40	L-2	3/18/77	97.1	0.028
99952-48	L-3	3/9/77	97.7	0.049
99952-41	K-3	3/3/77	91.0	1.6*
99952-42	K-4	3/3/77	79.4	7.9*
99952-43	K-5	3/3/77	48.7	interference

*Includes all amphibole minerals.

15152537

123Z00541

487269

GRACE

Construction Products Division

To: W. R. Hanlon

From: F. W. Eaton

Date: February 14, 1979

Subject: Sims Landmark Fertilizer-Air Sampling Results

Ref: FWE to M. DiB. memo dated 2/14/79

cc: M. DiBenedictis
H. C. Duecker
J. W. Wolter
E. S. Wood
J. C. Yang

03630739

One thing to be learned from sampling Landmark Fertilizer is that it is impossible to pre-judge the working environment and predict user exposure to tremolite fibers. Based on an initial survey of Landmark January 9, 1979, it was my opinion that with minor changes to the installed dust collecting system, fiber exposures would be less than the OSHA Limits. Since E. S. Woods April 13, 1978 Guidelines require exposures less than 2.0 and 10.0 fibers/cc without Engineering controls, Landmark was sampled January 18, 1979.

The first air samples were taken on two men charging approximately 80 bags of L-4 into the charging enclosure with the dust collecting system off. Dust generated during this operation was excessive and the plant manager, Lamar Steem, insisted that the dust collecting systems be turned on for the remainder of all sampling. As can be seen from the attached air sampling record sheet and TWA calculations, the two men charging vermiculite exceeded OSHA TWA and ceiling TLV's. It should be pointed out that on samples L-1 & 2 and L-10 & 11, the men were charging vermiculite at the side of the hopper enclosure. On Sample L-16 & 17, both men were inside the hopper enclosure with the dust pick-up point at their back. These tests also indicate considerable fiber exposure in handling empty bags from the charging station to and into the waste paper bailer.

In light of E. S. Woods memo February 2, 1979 concerning lightweight fertilizer use of Libby derived vermiculite, you should advise M. DiBenedictis of the possible options before submitting these results to Landmark.

The following is general information on Sims Landmark for the record. Landmark is primarily a field (heavy) fertilizer producer in and for Ohio. The Sims facility is Landmark's only ammoniated fertilizer plant but there are several other dry blend and seed plants in the state. Equipment and process flow at this facility is very similar to the ammoniated fertilizer process at Agway/Big Flats New York. The main difference is that this facility is much newer and there are three installed dust control systems with pick up points provided at most all dump and transfer points. All control devices are baghouses. Lawn fertilizer is handled by the Farm Supply Division of Landmark and prior to being manufactured at the Sims Facility, was private labeled by a producer in PA. According to Lamar Steen Plant Manager, lightweight fertilizer and pesticides represent a very small portion of Sims Landmark Total Production. 1979 production forecast is 620 tons lawn and 55,000 tons field fertilizer. The total 620 tons of lawn fertilizer is produced at

15102882

108Z00482

03630740

at one time, stored in a bin, and bagged as required. Although there are weed killers such as 2-4D, the bulk of light weight (lawn) fertilizer is 22-11-7 bagged in 20, 25, and 33 1/3 lb. bags. In the 22-11-7 formulation, there is 221 lbs. of L-4 vermiculite per ton of fertilizer. Total vermiculite required for 620 tons is approximately 5,600 - 4 cu.ft. bags. According to Steen approximately 100 production hours is required to produce the 620 tons.

F. W. Eaton

FWE/cc

Attachment

15102883

108Z00483

W 3/24/79

LANDMARK FERTILIZER 03630741

JAY — TRACTOR DRIVER

(1)	23	× 22.68	= 521.64	(FILTER LOST DURING SAMPLING)
(6)			=	
(7)	33	× 0.39	= 12.87	
(10)	23	× 4.09	= 94.07	
(12)			=	(FILTER LOST DURING SAMPLING)
(14)	26	× 0.66	= 17.16	
(16)	15	× 28.22	= 423.30	
	120		1069.04	

$TWA = \frac{1069.04}{120} \times \frac{7}{8} = 7.8$ f/cc

BILL CORY — BIN MAM

(2)	17	× 30.93	= 525.81	
(5)	53	× 0.73	= 38.69	
(11)	22	× 4.28	= 94.16	
(13)	29	× 0.88	= 25.52	
(15)	15	× 0.28	= 4.20	
(18)	26	× 7.23	= 187.98	
(17)	41	× 23.21	= 324.94	
	176		1201.3	

$TWA = \frac{1201.3}{176} \times \frac{7}{8} = 6.83$ f/cc

15102884

108Z00484

PAUL - SCALE MAM

(3)	61 ×	1.40	= 85.4
(9)	35 ×	1.95	= 68.25
	96		153.65

$TWA = \frac{153.65}{96} \times 7/8 = 1.4\ f/cc$

DOUG - FOREMAM

(4)	51 ×	0.50	= 25.5
(8)	43 ×	0.50	= 21.5
(19)	48 ×	0.98	= 47.04
	142		94.04

$TWA = \frac{94.04}{142} \times 7/8 = 0.58\ f/cc$

CONSTRUCTION
PRODUCTS
DIVISION

PAGE 1

REQUEST FOR TECHNICAL SERVICE

NUMBER: [illegible]
GROUP: AG/HORT
DATE: 1-19-79
CHARGE NO.: 70-984
REQUESTOR: F.W. EATON
MARKETING or MANUFACTURING APPROVAL
NAME: F.W. EATON
APPROVED: FE

03630743

PROBLEM TITLE:
DETERMINE % TREMALITE AG 1-4

SIGNIFICANCE:

SPECIFIC OBJECTIVE:
DATA COLLECTION TO COMPARE WITH PREVIOUS RESULTS & AIR SAMPLES TAKE AT LANDMARK FERTILIZER 1-18-79

SUGGESTED APPROACH:

DEADLINE (Last day information will be of value):
BY 2-2-79 IF POSSIBLE, ATTACH TO LANDMARK AIR SAMPLE (FIBER) RESULTS

DETAILS OF PROBLEM:
DETERMINE % TREMALITE IN AG 1-4 PRODUCED AT WILDERS - D-18 & USED AT LANDMARK FERTILIZER MAY CAUSED A IN THE PRODUCTION OF LAWN FERTILIZER

ACCEPTED BY RESEARCH DEPT.: [signature] DATE: 1/19/79
ASSIGNED TO: C. Wallock / J.P. Wallace / N. Catalfo
ADDITIONAL COPIES: Original to Library: H.C.Duecker, W.R.Hanlon, J.W.Wolter, E.S.Wood

CONFIDENTIAL

15102886

108Z00486

)UEST FOR TECHNICAL SERVICE

NUMBER: 67026
GROUP: Ag/Hort Lines
ACTUAL COST: $420.00
REPORTING DATE: January 30, 1979

03630744

SUMMARY and RESULTS:

The rock and tremolite fiber content of the AG L-4 used in the Landmark fertilizer were determined by C. Walloch and found to be 1.85 and 0.039%, respectively.

Even though the tremolite fiber content was quite low, the fibers appeared to be very fine, splintery, and well-distributed throughout the whole sample, which was in contrast of fine bundle and aggregates normally present in our products.

Eighteen air-fibrous samples were received and evaluated. Out of the group, eight samples exceeded the 2.0 fiber/ml limit. Again it indicated that an abundance of fine fibers was present.

Julie C. Yang

JCY:mlr

15102887

108Z00487

(1)

GRACE

AIR SAMPLING RECORD SHEET

PLANT LOCATION LANDMARK FERTILIZER / MT. GILEAD OHIO
CONTAMINANT FIBER
SAMPLING BY F.W. EATON
DATE: 1/18/79

SAMPLING CONDITIONS:
OUTSIDE COLD / INT. LIGHT SNOW
INSIDE DRAFT NO
VISIBLE DUST YES - BIN CHARGING AREA

HOUSEKEEPING:
FAIR - TYPICAL FERTIL. PLT.

→ FILTERS VERY DUSTY

03630745

Sample Number	Employee Name	Job Location and Description	Remarks	Pump Number	Pump Off	Pump On	Sampling Time	Flow Rate	Total Sampled Volume	Lab Evaluat
L-1	JAY	TRACTOR DRIVER	EMPTYING APPROX 80 BAGS L-4 INTO CHARGING HOPPER	5	1006	0943	23	1.6		22.6
L-2	BILL CROY	BIN MAN	SAME AS L-1	8	1003	0946	17			30.9
L-3	PAUL	SCALE MAN	WEIGHS BATCH MATL. IN SEMI ENCLOSED ROOM	6	1053	0952	61			1.4
L-4	DOUG	FOREMAN	COVERS ENTIRE PLT. OPERATION	7	1048	0957	51			0.5
L-5	BILL CROY		ODD JOBS		1058	1005	53			0.73
L-6	JAY		DUMPING SULPHATE IN HOPPER		—	1007	LOST	FILTER		
L-7	JAY		CHARGING HOPPER W/ MATL OTHER THAN VERMICULITE		1056	1023	33			0.39
L-8	DOUG	SAME AS L-4			1131	1048	43			0.5
L-9	PAUL	SAME AS L-3			1128	1053	35			1.95
L-10	JAY	SAME AS L-1	INCLUDING PUTTING EMPTY BAG IN BAILER		1119	1056	23			4.09
L-11	BILL CROY	SAME AS L-2	INCLUDING BAILING EMPTY BAGS.		1120	1058	22	✓		4.28
15102888										

Additional Comments: (1) SAMPLE L-1 & 2 TAKEN WITH DUST COLLECTION SYSTEM SHUT DOWN. MEN EXPOSED TO EXCESSIVE DUST IN 3 SIDED HOPPER ENCLOSURE. BECAUSE OF EXCESSIVE DUST ATMOS. SAID RESULTS WOULD NOT BE ACTUAL EXPOSURE AND REQUESTED INSTEAD THAT DUST COLLECTION SYSTEM BE TURNED ON FOR REMAINDER

Laboratory Evaluation By: Nancy Cataldo
Date: 1/29/79

(2)

GRACE

AIR SAMPLING RECORD SHEET

10820489

PLANT LOCATION LANDMARK FERTILIZER
CONTAMINANT FIBER
SAMPLING BY F.W. EATON
DATE: 1/18/79
— FILTER VERY DUSTY

SAMPLING CONDITIONS:
OUTSIDE
INSIDE DRAFT
VISIBLE DUST

HOUSEKEEPING:

03630746

Sample Number	Employee Name	Job Location and Description	Remarks	Pump Number	Pump Off	Pump On	Sampling Time	Flow Rate	Total Sampled Volume	Lab Evaluat
L-12	JAY		PUTTING EMPTY BAGS INTO BALER	8	—	1302	—	LOST FILTER		
L-13	BILL CROY		SAME AS L-12	5	1333	1304	29	1.6		0.88
L-14	JAY		CLEAN UP AROUND VERM HOPPER HAULING POTASH		1348	1322	26			0.66
L-15	BILL CROY		CHECKING BINS		1348	1333	15			0.28
L-16	JAY	SAME AS L-1			1420	1405	15			28.2
L-17	BILL	SAME AS L-2			1420	1406	14			23.2
L-18	BILL		BAILING EMPTY BAGS & COFFEE BREAK		1447	1421	26			7.2
L-19	DOUG	SAME AS L-4			1432	1344	48			0.9
L-20	ENGINEERING	SAMPLE ENCLOSURE WHILE DUMPING L-4	SAMPLE INSIDE HOPPER		1422	1407	15	↓		22.2
15102889			SAMPLE TAKEN DURING SAME PERIOD AS L-16 & 17							
	SAMPLING TESTS STOPPED APPROX 1500 AS PLANT WAS SHUT DOWN DUE TO PLUG UP OF COOLER DISCHARGE CHUTE									

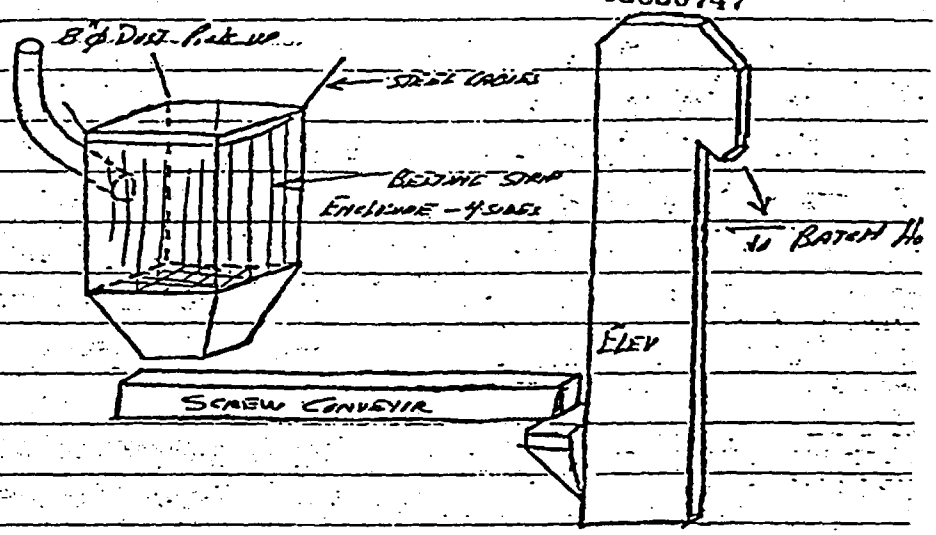
Additional Comments ② EQUIPMENT & PROCESS FLOW @ LANDMARK IS FAIRLY SIMILAR TO THE AMMONIATED FERTILIZER PROCESS AT AGWAY / BIG FLATS N.Y. IN ADDITION TO DUST CONTROL ON THE DRYER (BAGHOUSE), THERE

Laboratory Evaluation By: Nancy Catalde
Date: 1/29/79

ADDITION COMMENTS (JACOBSEN FERTILIZER)

(3)

03630747



TYPICAL CHARGING HOPPER

ON THE MAIN FLOOR OF THE FERTILIZER PLANT THERE ARE STORAGE BINS FOR RAW MAT'L & FINISHED PRODUCT PL THREE (3) CHARGING HOPPERS SIMILAR TO ABOVE SKETCH. BULK MAT'L IS CHARGED WITH FRONT END LOADERS (3) THROUGH BELTING STRIPS AROUND HOPPER HOLE/ENCLOSURE. CONSIDERABLE DUST IS GENERATED AS TRUCK GO UP & DOWN ISLE AND WITH WHICH DUMPED INTO HOPPER.

(4) APPROX 80 BAGS OF VERMICHITE ARE PLACED OUTSIDE HOPPER ENCLOSURE EACH TIME BATCH HOPPER IS FILLED. BELTING STRIPS ON ONE SIDE ARE FOLDED BACK AND MEN STAND IN ENCLOSURE ON GRATING WHILE EMPTING

BAGS OF VERMICULITE. MOST OF THE DUST IN THIS OPERATION IS SELF GENERATED ESPECIALLY BY JAY, A NEW YOUNG EMPLOYEE. THE SAME IS TRUE WHEN HE DRIVES FORK END LOADER.

(5.) FOREMAN COVERS ENTIRE OPERATION BUT USES AMMONIATOR CONTROL ROOM AS HEADQUARTERS ON THIS DAY (1-18-79) HE SPENT MAJORITY OF TIME BRUSHING & ADJUSTING SCREENS.

(6) SCALE MAN IS IN ROOM ADJACENT TO WEIGH HOPPER. THERE IS AN OPEN WINDOW NEXT TO WEIGH HOPPER.

(7) THERE IS 221 LBS 1-4 VERMICULITE PER TON BATCH OF FERTILIZER. ON 1-18-79 1ST SHIFT, THEY PRODUCED 34 T OR HANDLED 7514 LBS OF VERMICULITE APPROX 240 BAG (6000 LBS) OF 1-4 WAS CHARGED INTO THE BATCH HOPPER DURING THE SAMPLING PERIOD. NORMAL PRODUCTION RATE IS 6 T/HR.

15102891

108Z00491

457270

PRODUCTS DIVISION

41020

PAGE 1

GROUP: Pet Marketing Group

DATE: July 27, 1979

CHARGE NO.: X-6-293 580

REQUESTOR: R.A. Merther

MARKETING or MANUFACTURING APPROVAL:

NAME:

APPROVED:

ADMINISTRATIVE RECORD

REQUEST FOR TECHNICAL SERVICE

PROBLEM TITLE: Analysis of Monokote for Asbestos - Floyd County Board of Education

SIGNIFICANCE: School District needs to determine if Fireproofing Material contains Asbestos

SPECIFIC OBJECTIVE: To determine the type of material (MK-3 or MK-4) removed from Rome Georgia school

SUGGESTED APPROACH:

DEADLINE (Last day information will be of value): ASAP

DETAILS OF PROBLEM:

ACCEPTED BY RESEARCH DEPT.: DATE: 7/27/79

ASSIGNED TO: M. Doyle

ADDITIONAL COPIES: Original to Library; H.C.Duecker, R.A.Merther, L.S.Sh[illegible], B.A.Blessington

CONFIDENTIAL

15080806

103Z00764

[redacted] ACTUAL COST: $150.00

41021 REPORTING DATE: August 15, 1979

SUMMARY:

The fireproofing material removed from Rome, Georgia school was examined by x-ray diffraction analysis, chemical dissolution and microscopic examinations.

Chrysotile fibers were found to be present in appreciable quantities (≥5%). Thus, it was concluded that the material was MK-3.

EXPERIMENTAL:

1. Material Examined As Received

By X-Ray Diffraction Method

Pulverize the material to -100 mesh size in a SPEX mill and x-ray.

Major: Gypsum, Vermiculite

Minor: Quartz and Chrysotile (Suspected)

Microscopic Observation

Long fiberous material (100x) was shown in the matrix.

2. Calcination

The received material was crushed to -20 mesh then heated in a platinum crucible with cover for 16 hours at 500°c to burn off the organic or cellulosic fibers.

The remaining residue was examined by polarized microscope at 430x and found long thin fibers of chrysotile.

3. Acid Dissolution

One gram of the received sample was digested with hot 1 liter of 0.01 N HCl for about 1 hour. The mixture was cooled off and filtered through a 0.45μ millipore filter. The solid residue was dried and examined by light microscope. Most of the gypsum which adhered to the fibers was dissolved but the chrysotile fiber remained in such as dilate acid solution.

Light miscropic examination (430x) showed the presence of long thin chrysotile asbestos fibers with the characteristic optical properties, (index of refraction ect.). The estimated quantity of the fiber in the sample was larger than 5%.

REFERENCE:

X-Ray File Misc. 293
Notebook 651-13

Julie C. Yang

JCY:mgd

15080807

103Z00765

FLOYD COUNTY BOARD OF EDUCATION
1307 REDMOND CIRCLE
ROME, GEORGIA 30161
404/234-8228

BOARD MEMBERS
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WILLIAM H. BOLING
ASSISTANT SUPERINTENDENT
NEWTON A. WHATLEY
ASSISTANT SUPERINTENDENT

41622

July 23, 1979

Mr. Bob Merther
62 Whittemore Ave.
Cambridge, Mass. 02140

Dear Sir:

Please find enclosed a sample of material used in one of our schools.

We understand from the contractor that the material is "MONOKOTE".

The Georgia State Department said that the material contains asbestos and would have to be removed.

We would like for you to analyze this sample and let us know if it contains asbestos and if so what percent it contains.

We have been advised that the State requires a polarized light microscopic test. Alos, a dispersion staining test.

Also, if you have any information as to whether or not this will meet Environmental Protection Agency requirements for use in schools, we would be interested in having it.

Sincerely,

Bill Toles

Bill Toles
Director of Maintenance

BT/sjs

15080808

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487270

ZONOLITE
CONSTRUCTION PRODUCTS DIVISION

41015

W. R. GRACE & CO. 62 WHITTEMORE AVENUE CAMBRIDGE, MASSACHUSETTS 02140 617-876-1400

This is to certify that no commercial asbestos is used in the manufacture of MONOKOTE® 5. Further, any trace contaminants of naturally occurring forms of asbestos in MONOKOTE, are bound in the in-place MONOKOTE so as to prevent asbestos fibers from entering the environment.

15080805

GRACE

103Z00763

4/87282

J. C. Yang

CURRENT ACTIVITIES OF ZONOLITE® ORE RESEARCH GROUP

02225313

PRIORITY I:

a) Assist Libby to determine % tremolite fiber in bag house dusts, etc.

b) Assist Libby to determine % tremolite fiber in samples collected at various stages of mill operation (sampling at Libby in process).

c) Evaluate samples from North Carolina State University, using different amines, tremolite depressant, and high pH media for tremolite removal (materials balance).

d) Generate basic information on vermiculite for horticultural applications and support on other research activities such as Bark Ash patent work.

PRIORITY II:

a) Support and evaluate all the material in the engineering projects on fiber removal:

   1. Binder development problem
   2. Salting program
   3. Electrostatic spray test
   4. Fluid bed tests

b) Laboratory evaluation of air-elutriation method for fiber removal and other techniques.

c) Count all the air samples for tremolite fiber content, collected in all CPD operations (mines, plants, job-sites, etc.) and, also, when working with products.

d) Support and count fibers in all the samples collected in the Control drop test.

e) Determine total dust content in operating areas.

f) Determine the asbestos fiber and silica content in MONOKOTE from California.

J.C.Yang:mlr
2/4/77

Continued.....

15025910

CURRENT ACTIVITIES OF ZONOLITE® ORE RESEARCH GROUP
(continued)

02225314

PRIORITY III:

a) Osmotic swelling and delamination of vermiculite from thick booklets to thin individual flakes which will result in better dispersion in process where mica can be used.

b) Check the variability of amines used in flotation agent received from vendor for Enoree operation (WRC).

c) Differentiate the hydrobiotite and vermiculite in our ore makeup in physical and chemical properties which may give us a better understanding for expandibility and ion release in horticultural applications.

d) Develop reproducible and accurate methods for tremolite fiber determinations as required.

e) Compile information on quantitative silica content of all the plaster of Paris from all the plants manufacturing MONOKOTE products. The determinations will be made several times a year.

f) Communicate and follow-up with Dr. W. Smith, Health Research Institute, Fairleigh-Dickinson University, on Animal Studies.

g) Verify Libby counting data every two months.

h) Communicate with outside agencies, institutions, on similar health problems and keep up-to-date developments and state-of-art in instrumental analysis.

i) Plan to join a nationwide Proficiency Analytical Testing program (PAT) sponsored by NIOSH to evaluate in-house technique and accuracy for analyzing asbestos fibers. Recently, a statistical study of in-house counting accuracy was made.

Julie C. Yang:mlr
2/4/77

15025911

088Z00949

Arthur D. Little, Inc. ACORN PARK · CAMBRIDGE MASSACHUSETTS 02140 · (617) 864-5770

January 31, 1973

Dr. Julie C. Yang
Senior Group Leader
W. R. Grace & Co.
Rock Processing Chemicals
Construction Products Division
Cambridge, Massachusetts 02140

Dear Julie:

Per your letter of January 11, 1973, and subsequent telephone conversations with Dr. Arnold Rosenberg and you, I am enclosing a report providing an analysis of asbestos (tremolite and actinolite) content of seven (7) unknown samples as well as an operating procedure for determining asbestos content in Monokote samples. The method employed has demonstrated a 2σ confidence minimum detectable limit of 0.15 weight percent, which I think is especially good for a procedure based upon X-ray diffraction methods.

The overall cost for this work, which we will bill to your P. O. No. 41574, is $1800. This includes the small carryover from the previous task (December 19, 1972), diffraction scans of 12 samples, Method B and C point count data for seven (7) samples, Method C point count data for an additional seven (7) samples, and finally, specification of a measurement procedure.

I have retained all submitted samples and prepared X-ray samples should they be required in the future.

Very truly yours,

Edward T. Peters

/mc

Enclosure

January 30, 1973

PROCEDURE FOR MEASURING ASBESTOS CONTENT OF MONOKOTE MIXTURES FOR W. R. GRACE & CO.

03641134

SUMMARY

An X-ray diffraction procedure has been developed for determining the presence of tremolite and actinolite forms of asbestos in commercial mixtures of vermiculite and gypsum, such as monokote. Based upon the results from known chemistry standards, asbestos can be identified in these products with a 2 σ minimum detectable limit of 0.12 weight percent for tremolite and 0.15 weight percent actinolite. Of the seven samples submitted for measurement of asbestos content, all were found to have less than the minimum detectable limits of asbestos, with the following exceptions: African #3 - 1.90% tremolite and Kearney #3 - 0.30% tremolite.

INTRODUCTION

In December 1972, members of the Construction Products Division, W. R. Grace, Inc., reviewed with us a need for accurately determining the asbestos content of various commercial product mixtures, such as monokote. It was agreed that X-ray diffraction analysis appeared most practical. Analysis of several standards (0.5, 1.0 and 2.0% tremolite in monokote) revealed that the presence of asbestos could be detected by a diffraction scan strip chart recording. To explore the possibility of improving the sensitivity of the X-ray method, Arthur D. Little, Inc., conducted a second set of experiments based upon the fixed count X-ray method. This proved successful, providing a minimum detectability limit of 0.12 weight percent tremolite in monokote. These results were presented in our report dated December 19, 1972.

On January 11, we were asked to:

1) Prepare a calibration curve for the quantitative analysis of actinolite, utilizing

   a) Fixed count procedures, as before.
   b) Area under the curve, after slow scans.

2) Conduct an analysis of several expanded vermiculite samples and of the monokote product prepared at various locations from Libby ore to determine tremolite and actinolite content.

In our preliminary work, it became clear that the actinolite standard mixes were different than the other samples, in that they resisted dispersion in mixing with amyl acetate. Subsequently, it was determined that the standards were improperly prepared and a new set was submitted. On January 26, 1973, a final expanded vermiculite sample was submitted for analysis. This did not have the same pre-treatment as the other samples, resulting in a much coarser particle aggregate size.

15034515

Arthur D Little Inc.

092Z01580

Samples were prepared and analyzed similar to the earlier work. A description of the methods used and procedural outline are presented.

EXPERIMENTAL PROCEDURE

It was assumed that the pre-treatment provided by W. R. Grace resulted in uniform, well blended samples. X-ray samples were prepared by mixing 20-50 mg of the powder mixture with amyl acetate to make a slurry. Thorough mixing was carried out in a mortar and pestle after which the slurry was poured onto a glass microscope slide and dried. All X-ray diffraction data were carried out with a copper X-ray tube operated at 40 kV and 20 ma. The apparatus utilized a post beam monochromator equipped with a graphite crystal to minimize scattered, background radiation. Based upon the previous study and upon information from diffraction scans of the monokote mixtures and pure asbestos standards, it was determined that the most suitable diffraction line positions for the asbestos peaks free from interference from other peaks were at:

Tremolite - $2\theta = 28.5°$
Actinolite - $2\theta = 12.4°$ and $28.5°$

X-ray data were collected according to three basic procedures, as follows:

A. Diffractometer Scan

Scanning was conducted at a rate of 1°/minute over the range of $2\theta = 4-50°$. This scan exhibited all diffraction peaks. As shown earlier, this approach permitted detecting the presence of 1% tremolite (at $2\theta = 28.5°$), and from the present work, 1/2% actinolite (at $2\theta = 28.5°$). As both peaks occur at 28.5, the direct scan approach can only say one or the other or both forms of asbestos are present in monokote in excess of 1%.

B. Area Display (Slow Scan)

Scans were made at 1/4°/minute over the range of interest ($2\theta = 11.9 \rightarrow 12.9$ and $28.0 \rightarrow 29.0°$). As there was little "area under the curve" for most samples, equivalent data were collected by measuring counts in 60 seconds at 28.0 and 29.0° as background and counts in a 120 second scan over the peak from 28.0° to 29.0° as peak. The signal is then taken as peak minus background.

C. Point Count

Data were collected at fixed positions from peak (12.4 and 28.5) and background (11.9 and 28.0), recording the time (seconds) to collect 6400 counts, providing a 2σ probable error of 1.68%. In the case of the expanded vermiculite samples, the 28.5° area of interest was influenced by the tail of an adjacent, broad peak; for these samples, background was taken to be the average of measurements at 28.0 and 29.0°.

EXPERIMENTAL RESULTS

Diffraction scans for the 12 submitted samples are attached. Examination of the traces showed the expected peaks in all cases. The three expanded vermiculite samples all showed variation from one another, which is attributed to small differences in the composition of the ore or processing variables. The scans of monokote prepared at four locations were essentially identical. The results from the various methods of analysis are given below, with measured data presented in Table 1:

Method A

From the standards, a peak at 28.5° is observed with as little as 0.5% actinolite or (from the previous work) 1.0% tremolite. However, at least 2.0% actinolite is required to observe the peak at 12.4°. Based upon the higher backgrounds and interfering tails of adjacent peaks present in the monokote and expanded vermiculite samples, it is concluded that diffraction scans are suitable for identifying the presence of asbestos in quantities of 2 weight percent or greater.

Method B

As can be inferred from the data presented in Table 1, slow scanning fails to exhibit a peak distinguishable from background. Using the more exacting measurement of counts collected at background (120 sec at 11.9° and 120 sec at 12.9° or 120 sec at 28.0° and 120 sec at 29.0°) and from background plus peak (240 sec for scan from 11.9→12.9° and 28.0→29.0°), one observes in Table 1 that background is generally higher than peak count. Although no clear explanation can be provided for this, it is assumed that background is not uniform over the range scanned. The fact that peak signal is so low, precluding a measurable area above background, rules out this approach for determining asbestos content in monokote samples.

Method C

Experimental data collected according to the Method C procedure are presented in Table 1. Each measurement is converted to a counts/second basis, with appropriate correction for the difference in background counting rate at P and B positions as determined from the monokote blank. A plot of signal (i.e., peak less background) versus composition for the various standards is presented in Figure 1. With the exception of one datum point, the tremolite data is in excellent agreement with the earlier calibration curve (December 19 report), giving considerable credence to the experimental approach that has been employed.

The actinolite data show some scatter. The curve at 12.4° (with a 2σ - confidence, minimum detectable limit of 0.15%) is employed to identify the presence of actinolite. From Figure 1, the corresponding count rate for the 28.5 actinolite peak is determined and subtracted from the corrected 28.5° signal. Any remaining signal is attributed to tremolite.

Experimental data for the seven unknown samples is also presented in Table 1. As the 28.5° peak position occurred on the tail of a major expanded vermiculite peak, a more appropriate background was obtained by averaging data collected at 28.0 and 29.0° positions. Even this approach resulted in an over-correction for background. This high background difficulty is the result of a broad expanded vermiculite peak being present at 26.7° in the ~~North~~ Carolina ore employed for standards, where there was no interference from peak tails at 28.0°, whereas it is present at 27.3° in the Libby ore resulting in a peak tail at 28.0°. As a consequence, the absence of a peak at 28.5° in the four monokote samples using Libby ore was inferred by a measured peak to background ratio of 0.86 for all four samples.

The three expanded vermiculite samples showed no signal at 12.4°, precluding the presence of actinolite. A measured signal at 28.5° was therefore attributed to tremolite corresponding to 1.95% and 0.30% for the African #3 and Kearney #3 samples, respectively.

RECOMMENDED PROCEDURE

Based upon the experimental results described above, the following procedure is recommended for determining the presence of asbestos (actinolite and/or tremolite) in monokote samples:

1. Mix 20 to 50 mg monokote mix with 20 to 30 drops amyl acetate, mix in mortar and pestle, pour onto a glass slide (covering an area of 4-6 cm^2), and allow to dry.

2. Employing a Philips vertical diffractometer equipped with a copper target X-ray tube operated at 40 kV and 20 ma, 1° divergence slit, 0.001 inch receiving slit and graphite-crystal post beam monochromator,* collect the following data:

   a. Measure time to collect 6400 counts at $2\theta = 12.4°$ and convert to counts/second = P1

   b. Measure time to collect 6400 counts at $2\theta = 11.9°$ and convert to counts/second = B1

   c. Calculate S1 = P1 - B1

      1. If S1 = 0.4 or lower, assume no actinolite is present.
      2. If S1 > 0.4, read % actinolite from curve (1), Figure 1. Also, read counts/second at same % actinolite from curve (2) and call S2.*

   d. Measure time to collect 6400 counts at 28.0, 28.2, 28.5, 28.8 and 29.0°; convert to counts per second; plot counts/second versus 2θ; draw smooth curve through points at $2\theta = 28.0$, 28.2, 28.8 and 29.0°; take difference between 28.5° point and the smooth curve and call P2.

*Other experimental apparatus could of course be used, but would probably require new calibration curves.

15034518

e. Calculate S2 = P2 - S2*

1. If S2 = 0.7 or less, assume no tremolite is present.
2. If S2 > 0.7, read % tremolite from curve (3), Figure 1.

3. For procedure as presented above, the minimum detectable limit of tremolite is 0.12 and actinolite is 0.15 weight percent, respectively.

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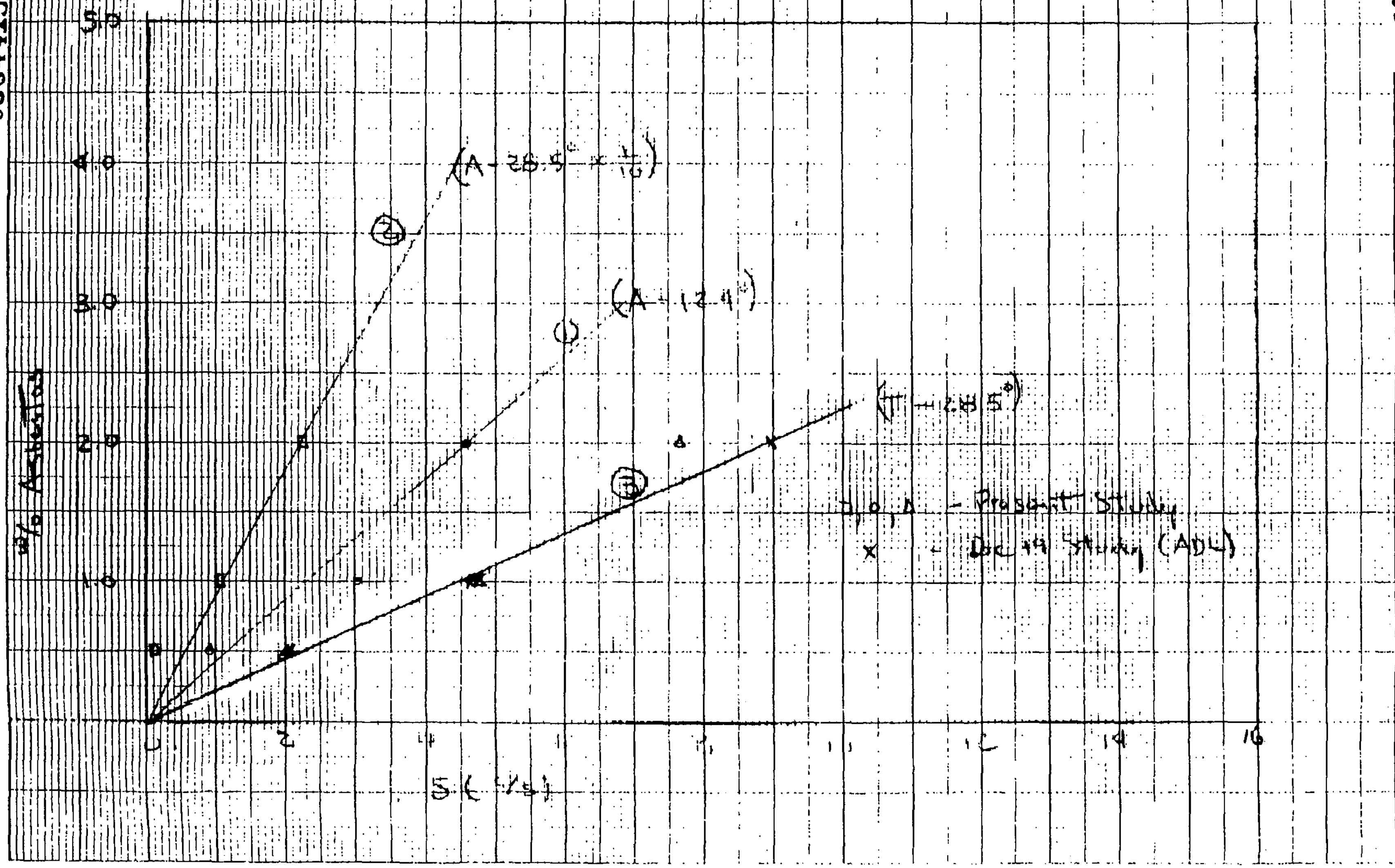
FIGURE 1

CALIBRATION CURVE FOR
ASBESTOS IN [illegible]



15034520

CAMBRIDGE

TO: O. F. Stewart, Enoree, S.C. DATE: August 31, 1973

FROM: Julie C. Yang

SUBJECT: Tremolite Determination in South Carolina Vermiculite Ores

CC: R. M. Vinsing
H. A. Brown
T. Lyall
W. F. McCord
J. L. Wright
H. C. Duecker
A. M. Rosenberg

FILE: 150 - Asbestos Determination in Vermiculite Ores

TABLE OF CONTENTS:

OBJECTIVE:

To determine the tremolite content in S.C. vermiculite ores at various locations.

SUMMARY:

Instrumental means were employed to determine the asbestos content in S.C. ores. The findings are listed as follows:

1. There are asbestiform fibers present in S.C. ores, but mostly very fine and small. All the identifiable fibers are hornblende, an aluminum-rich amphibole. So far no information has been published to indicate whether this material is detrimental to health or not, as of other types of amphiboles (tremolite, crocidolite and amosite) and chrysotile asbestos.

2. The only detectable difference between Allen and Waldrup type specimens from the same location (Poole #7) is that the Allen type has relatively higher talc and hornblende contents than the Waldrup sample.

MATERIALS RECEIVED:

The following samples were submitted from O. F. Stewart, Enoree, S.C. at R. M. Vinning's request for tremolite analysis:

Sample No.	Deposit	Type
122210-1	Poole #7	Allen
2	Poole #7	Waldrup
3	Allen	Allen
4	Poole #3	
5	Burns	Waldrup
6	Meadows	Waldrup
7	Yarborough	Waldrup
8	Lanford	Waldrup

EXPERIMENTAL WORK:

Instrumental Methods Employed

X-Ray Diffraction	A rapid, non destructive method to identify crystalline materials such as mineral species
Scanning Electron Microscope (SEM)	To study the morphology of particules
Optical Microscopy	To identify individual particules by its optical properties
Electron Probe Analysis (EPA)	To analyze the elements present and the relative quantities of them

DISCUSSION OF RESULTS:

The instrumental analyses were done at Arthur D. Little, Inc. by Dr. E. Peters and his colleagues, the interpretations were made by J. C. Yang in collaboration with E. Peters.

1. X-Ray Diffraction Data

The ore samples as received showed an intense x-ray diffraction peak at 20-28.5°. The position employed for our previous quantitative measurement of tremolite in Monokote® and expanded vermiculite from Libby mine. Attempts were made to expand the vermiculite 1) chemically with conc. hydrogen peroxide and 2) thermally for 3 minutes at 1400°F, but in the expanded samples the unwanted peak at 28.5° persisted. It

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was then decided to perform the x-ray diffraction analysis to provide identification and quantitative estimates of the mineral species present, and to examine the fibers in several samples by scanning electronmicroscope. If fibers were present, they would be identified by the optical properties and electron probe analysis for its elemental ratio.

The mineral species in the samples were analyzed and tabulated in Table 1.

All the samples were found to have x-ray diffraction peaks that correspond to hornblende or tremolite. Based upon the diffraction peak at 8.40 Å hornblende line, the quantity of hornblende in various samples were estimated and corporated with other results in Table 1. The distinction between hornblende and tremolite, as well as whether they are in platy or fibrous form was then determined by SEM and EPA.

2. Scanning Electron Microscopic (SEM) Examination and Electron Probe Analysis (EPA)

A few representative photographs and profiles for the elements present are shown in Figs. 1 to 5.

Elements corresponding to various peak position are:

Mg	1.25	K	3.30	Ti	4.55
Al	1.47	Ca	3.70	Fe	6.40
Si	1.75	Ca	4.05	Fe	7.05

Several fibrous particles were observed in each sample by SEM. The chemical composition by the probe analysis showed that the elemental ratio of 5Mg-1Al-16Si-4Ca, whereas the tremolite standard from Libby yielded a relative ratio of 5Mg-20Si-1K-3Ca. It thus appeared the analyzed fibers were hornblende instead of tremolite as we suspected. Other non-fibrous particles showed the typical ratio of 2Mg-1Al-5Si (Vermiculite) with occasional replacement of some or all of the Mg by K (hydrobiotite).

3. Optical Examinations

Two of the samples 22210-2, Poole #7 (Waldrup) and 22210-6, Meadows (Waldrup) were examined by optical microscope, utilizing the Montana tremolite sample as a standard. Observed fibers were found to have the refractive indices in the range of those of hornblende (measure γ_D= 1.623, 1.648) which are considerably higher than the values for tremolite (γ_D= 1.599, 1.625).

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4. Chemistry of Hornblende

Hornblende series is a family in the amphibole asbestos group which shows a considerable variation in composition. The principle features of the composition are the presence of both Ca and Na or K, the former dominating, the Al is partially substituted for Mg and Si, and most of the members are deficient in silica. The composition of the series may be expressed by the two end members:

$Ca_4Na_2Mg_{10}Al_2Si_{14}O_{44}(OH, F)_4$ edenite

$Ca_4Na_2(Mg, Fe)_8Al_6Si_{12}O_{44}(OH, F)_4$ hastingsite

Compared with tremolite $Ca_2Mg_5Si_8O_{22}(OH)_2$.

Fluroine commonly enters into the composition to replace OH in part, Mg by Fe^{II}, Al by Fe^{III}, as do Ti and Mn for cations.

Such as Pargasite $(Na,K)\ Ca_2Mg_4Al_3Si_6O_{22}(OH)_2$

and Barroisite $(Ca,Na)_{2.26}(Mg,Fe,Al)_{5.15}(Si,Al)_8O_{22}(OH)_2$

are the intermediate members.

These minerals are monoclinic in crystal structure, and have fiber-like appearance but usually very chunky (low aspect ratio). These are soft and easily pulverizable like clay minerals, and quite different from either the harsh Mg-amphibole, tremolite; Fe-amphibole, crocidolite. and Fe-Mg amphibole, amosite or from the silky, flekible chrysotile asbestos fibers.

No known literature published to date has been found discussing the effect of hornblende to health.

CONCLUSIONS:

1. All the S.C. ore samples analyzed are very similar in mineral compositions. All of them are rich in vermiculite and hydrobiotite with minor amounts of hornblende, talc and quartz.

2. All samples examined contain asbestiform fibers that have been identified as hornblende, an Al-rich amphobile, which are fiber-like under light and electron microscope. This material is, at this time, an unknown health hazard.

3. The difference of Allen & Waldrup type specimens from the same location is the relative talc and hornblende content. Allen type seems to be richer in both.

4. The SEM preparation procedure tends to emphasize the smallest size particles, so that the method is not quantitative. Of all these samples examined, 22210-2, Poole #7 (Waldrup) appeared to contain the largest fraction of fibers.

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RECOMMENDATIONS FOR ADDED STUDY:

Since hornblende is a fibrous amphibole, the carcinogenic potential of this material has not yet been found in any literature, but it may be questioned in the future because of composition and structure closely associated with tremolite and other amphiboles. It is suggested to contact outside agencies to have an animal study made on the potential carcinogenic effects of horblende, compared with tremolite and chrysolite fibers. Usually, the test will take one to two years, but by then we will know for sure whether this material exhibits any cancer-inducing potential.

Agencies and Institutions equipped to do this type of work:

1. Huntingdon Research Center.
   Huntingdon, England

   - where the carcinogenic screening of vermiculite (S.A.) was done in 1970-1972.

2. Dr. Lewis J. Cralley
   Occupational Health Program
   National Center for Urban and Industrial Health
   Public Health Serivce
   1014 Broadway
   Cincinnati, Ohio 45202

   - previous contact, had studied amphiboles and chrysotile of various length, pure synthetic chrysotile and chrysotile with added Ni, Fe, Co, etc. in the structure.

3. Dr. Paul Gross M.D.
   Industrial Hygiene Research Unit
   Dept. of Occupational Health
   Graduate School of Public Health
   University of Pittsburgh
   Pittsburgh, Pa. 15213

   presently Medical School Univ. of S. Carolina

   - previous contact, did similar studies as Dr. Cralley.

Julie C. Yang

Julie C. Yang

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Julie C. Yang
8/31/73

TABLE 1 DETERMINATION OF MINERAL COMPONENTS PRESENT IN SOUTH CAROLINA VERMICULITE ORE (BY X-RAY DIFFRACTION METHOD)

SAMPLE NO.	DESCRIPTION	VERMI-CULITE	HYDRO-BIOTITE	HORNBLENDE	TALC	QUARTZ
22210-1	Poole #7 (Allen)	+++	+++	++ (10-20%)*	++	+
2	Poole #7 (Waldrup)	++	+++	+ (2-5%)	?	Trace
3	Allen (Allen)	+++	+++	+ (5-10%)	+++	-
4	Poole #3	+++	+++	+ (2-5%)	++	+
6	Meadows (Waldrup)	++	+++	++ (10-20%)	-	Trace

Legend:

+++	Major	(>50%)
++	Intermediate	(10-40%)
+	Minor	(1-9%)
?	Doubtful	(<1%)
-	None	

* The percentage of hornblende in the parenthesis was determined by the intensity of $2\theta = 8.40$ A°.

Nos. 5, 7, 8 not determined.

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123Z00521

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CONFIDENTIAL

CAMBRIDGE

ADMINISTRATIVE RECORD

TO: H. C. Duecker

DATE: February 23, 1976

FROM: Julie C. Yang

SUBJECT: Libby Ore Evaluation - Ore Impurities

CC: H. A. Brown
J. W. Wolter
R. L. Oliverio/Libby
R. J. Kujawa/Libby
G. G. Vaplon/Libby
O. F. Stewart/Enoree
R. H. Locke
J. L. Young
File: 71-048

03627800

PURPOSE

The objective of this investigation is to determine the tremolite content for each of the three mill circuits and end products at Libby.

SAMPLE SELECTION

Samples have been collected by G. Vaplon:

(a) material which entered the circuit,
(b) material which came out of the circuit,
(c) screened plant products as control and comparison with (a) & (b).

Fourteen materials were received:

(1)	Clean Conc.	8 x 20	
(2)	Rough Conc.	8 x 20	
(3)	Rough Conc.	20 x 65	
(4)	Clean Conc.	20 x 65	
(7)	Rough Feed	8 x 20	
(8)	Clean Feed	8 x 20	
(9)	Rough Feed	20 x 65	
(10)	Clean Feed	20 x 65	
(11)	#1 Composite		
(12)	#2 Composite		
(5)	#3 Composite		
(6)	#4 Composite		
(13)	#5 Composite		
(14)	Humphrey Sizer Concrete	12/3/75	9:00 a.m.

EXPERIMENTAL

I) Humphrey Sizer

1. Separation

The rock and fiber were separated from the vermiculite plates by hand-picking.

2. Method of Analysis

Each portion has been weighed carefully and then x-rayed for their mineral content.

20152820

To: H. C. Duecker
From: Julie C. Yang
Feb. 23, 1976

Libby Ore Evaluation - Ore Impurities

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3. Results and Accuracy

	Wt. %	Accuracy ... %	
Vermiculite	86.71	86.71 ± 0.43	approx.*
Rock	10.58	10.58 ± 0.05	approx.*
Tremolite	2.71	2.71 ± 0.01	
Total	100.00		

*The rock content may be higher than the figure shown at the expense of vermiculite, since some of the granules can be classified as vermiculite fine aggregates (showed vermiculite x-ray pattern) but may not be expandable as we previously found (report 11/3/75 - Properties of Libby Vermiculite Ore). The fiber portion showed a good x-ray pattern of pure tremolite with no rock contaminations.

II) 8 x 20 Circuit and End Products #1, 2 & 3

1. Separation

The samples in this group were sized by Ro-Tap screening to +50 and -50 fractions. 100 gram vermiculite sample was Ro-Tapped for 16 minutes total, a ten minute increment first, then three 2-min.consecutive intervals to insure the achievement of constant weights.

Then from the +50 size fractions, fibers were hand-picked and weighed. The bulk materials remaining were then chemically expanded with 30% H_2O_2 individually. The light expanded vermiculite thus was separated from the heavy rocks and fiber bundles by water flotation. Both portions were collected, dried and weighed, then ground to -100 mesh and subjected to x-ray examination.

2. Method of Analysis

Tremolite remaining in the samples was determined by quantitative x-ray diffraction analysis, and the values were added to those of the hand-picked tremolite. In quantitative x-ray analysis a calibration curve was constructed to determine tremolite by adding a known amount of Libby tremolite (hand-picked from #2 composite, opened and cleaned) to a hand-picked pure vermiculite sample. The curve was made for determinations up to 10% tremolite.

The total area under the $2\theta = 28.5°$ in the diffraction pattern, the peak responded to the max intensity peak of tremolite, was computed for the quantitative studies, and a second peak (height only at $2\theta = 10.5°$) was employed as a check for the interference (Figure 1).

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3. Result and Accuracy of Analysis

Experimental results are listed in Table 1. Since the detection limit of tremolite by x-rays is about 0.2% in a specific sample, for very low concentration occurence tremolite has to be concentrated in the sample by removing the bulk of vermiculite. Vermiculite can be removed easily by chemical expansion with 30% H_2O_2 followed by flotation.

On the chart, three tremolite contents (actually the range) were given based on the detection limitations.

4. Comparison of Material from 8 x 20 Circuit and End Products Composite #1, #2 and #3

The rock content of composites #1 and #2 are in line with those of the concentrates in the 8 x 20 circuit, but the tremolite content in these composites are definitely higher than the concentrates. The exceptionally high tremolite content is noted in Composite #2. The fiber contents in the 8 x 20 concentrates are slightly less than those in the corresponding feeds.

III) 20 x 65 Circuit and End Products Composites #4 and #5

1. Separation and Analysis

The samples in this group were sized by Ro-Tap screening to 3 fractions, namely +70, -70 +100 and -100 mesh size using the procedure described in Section II 1.

In the +70 fraction of rough and clean concentrates, the fine fibers present were balled up to pea-sized white balls, which were separated by gentle screening. The fiber balls were retained on a 50 mesh screen and weighed. To check the fiber content, the weighed fiber balls were broken and redistributed in the sample and subjected to quantitative x-ray determination. Since the fiber contents were very low, vermiculite in these samples were expanded chemically and then removed by flotation prior to x-ray analysis.

In the -70 +100 and -100 mesh fractions, tremolite was determined directly from the sample as received; since the vermiculites present in these sizes are fairly small, the expansion and flotation will not separate the material effectively.

To: H. C. Duecker
From: Julie C. Yang
Feb. 23, 1976

Libby Ore Evaluation -
Ore Impurities

03627803

2. Results and Accuracy

The experimental data is presented in Table 2. In the +70 fraction of the rough and clean concentrates, the white fiber balls of tremolite separated by gentle screening were found to be 0.32 and 0.22% respectively, in comparison to the values of <0.34 and 0.25% by x-ray method.

Again the tremolite contents were given in a range (max. and min.) in Table 2, based on detection limitations of the method.

3. Comparison of Material for 20 x 65 Circuit and End Products #4 and #5

The fiber contents in the concentrates of the 20 x 65 circuit are definitely less than those in the corresponding feeds, and also in line with the end product #4 composite.

End product #5 showed quite a high fiber content (~3.5%) and also a high rock content. For a rough estimate, the unexpanded material in this composite is close to 40% of the total.

OBSERVATIONS and COMMENTS

1. In the 8 x 20 circuit and the end product #1 and #2, most of the fibers present are in heavy bundles and very small amounts of fine fibers except some adhered to the surface of the vermiculite platlets.

2. In the 20 x 65 circuit, most of the fibers present are opened fibrils or smaller bundles. They tend to ball up into small white spheres while the sample is being sized by screening.

3. In the end products #4 and #5, the fibers are too short to form balls but are distributed widely throughout the matrix.

4. From Tables 1 and 2, the concentrates in both circuits showed relatively less fiber than in the feeds.

5. The expansion of vermiculite followed by flotation is a good method for separating the vermiculite from the rocks and the fiber, and the fiber content is then determined by x-rays but the method is good only when the vermiculite size is reasonably large (~ 70 mesh or larger).

6. For the small sized vermiculite samples, the tremolite content can be determined only from the sample directly by x-rays quantitatively. If the need ever came to determine the rock content in the vermiculite, chemical delamination method with 15% LiCl can be employed. The method has been described in a previous report (T&A 48522, 9/12/75).

20152823

To: H. C. Duecker
From: Julie C. Yang
Feb. 23, 1976

Libby Ore Evaluation - Ore Impurities

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CONCLUSIONS

1. The possible tremolite content of end products of each size and of concentrates from the three circuits are:

Circuit	Tremolite Contents, percent Range	Mean
Humphrey Sizer	2.70 - 2.72	2.71
8 x 20		
Rough concentrate	0.21 - 0.71	0.46
Clean concentrate	0.10 - 0.59	0.35
20 x 65		
Rough concentrate	0.4 - 0.86	0.63
Clean concentrate	0.74 - 1.20	0.97
End Product		
Composites #1	1.67 - 2.17	1.92
#2	4.72 - 5.22	4.97
#3	0.41 - 0.89	0.65
#4	0.52 - 1.00	0.76
#5	3.45 - 3.97	3.71

2. Based on the experimental data, the approximate amount of tremolite present in tons per day, out of each of the three circuits, will be as follows:

Circuit	Total Materials out of the circuit (tons/day)*	Mean Tremolite Content (tons/day)
Humphrey Sizer	220	5.96
8 x 20	295	1.16
20 x 65	513	4.10

*based on 22 hours in a day.

3. The #2 composite showed the highest tremolite content (even more so than #5), and the fibers present are mostly in heavy bundle form, visible to the eye. This fact is also true for the material in the 8 x 20 circuit and other coarse end products #1 and #3. The tendency of fiber balling in the 20 x 65 circuit shows that the fibers are more opened or in thinner bundles in addition to some extra fines distributed throughout the end products #4 and #5, which will lead to the belief that there is some degree of down screening.

20152824

To: H. C. Duecker
From: Julie C. Yang
Feb. 23, 1976

Libby Ore Evaluation - Ore Impurities

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4. For quantitative x-ray analysis, the detection limit is about 0.2%. Because of the variation in x-ray response from sample to sample (variables such as orientation, sample thickness, and packing conditions), the accuracy of these determinations is approximately ±0.5% of the tremolite present. Therefore, in Tables 1 and 2, computations showed the maximum and minimum tremolite content possible to be present in the sample.

5. A previous report on Libby vermiculite and tremolite density determinations (1/13/76) showed an appreciable density difference between tremolite (2.92 - 3.1) and vermiculite (2.28 - 2.61 depends on the degree weathering) and the difference in morphology. Tremolite can be separated from vermiculite by air elutriation technique based on the difference in velocity of particle settling. Meanwhile, the vermiculite plates can be "polished" by removing some of the fine dust and fiber adhered to the surface in the air stream. A separate report will be written to describe the details of that aspect shortly.

6. The conclusions reached assume the samples are all representative samples of the operation. In reality, we know we have considerable variation in feed quality from minute to minute, hour-to-hour, and certainly from pile-to-pile. This experiment should be repeated to obtain a better feel for this variation. The sampling technique is probably the most significant problem in the study. Reasonably good analytical results can be obtained although very time-consuming. About $2.0M of laboratory time will be required to repeat this test.

Julie C. Yang

JCY:mlr

Attachments

20152825

Fig 1 X-Ray Diffraction Method

Calibration for Tremolite Determination

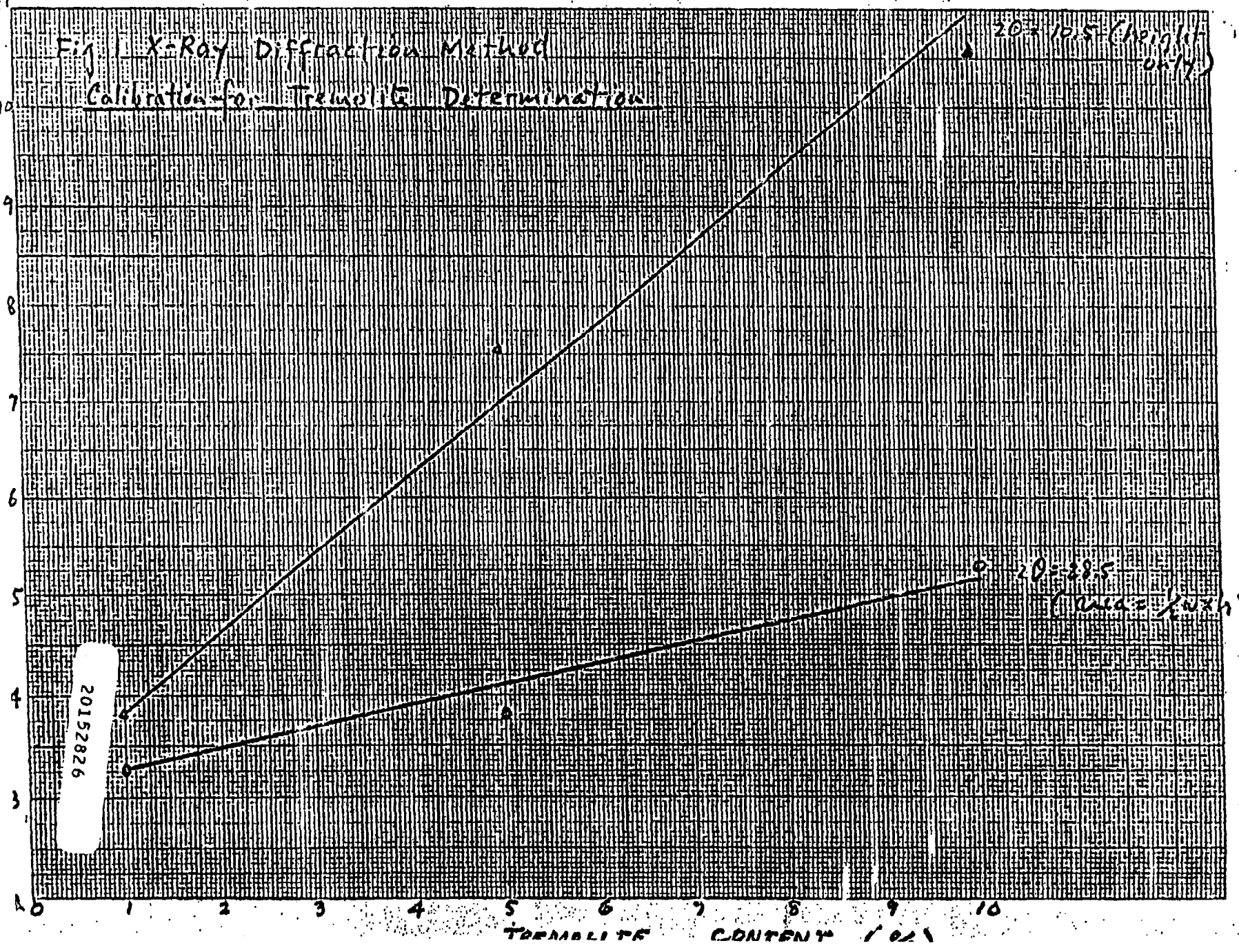

TABLE 1 Libby Ore Evaluation – 8x20 Circuit 4 Coarse Composites (#1, #2 + #3)

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Sample No.	Circuit	Wt (%)	+50 mesh Breakdown (%)	Obs.	−50 mesh Wt. (%)	−50 mesh Breakdown (%)	Total Tremolite Content min[a] (%)	Int.[b] (%)	max (%)[c]
7	Rough Feed 8X20	99.88		Very low T <0.5%	0.12	Def. by X-rays V = 0.05 ; T = 0.07	0.5	0.7	1.0
8	Clean Feed 8X20	99.53		Low T <1%	0.47	R+V = 0.39 T = 0.08	1.0	1.2	1.5
2	Rough Conc. 8X20	99.05	Exp. Verm.* 83.21 Rock+Trem.** 15.84	†T ≅ 0.16%	0.95	R+V = 0.90 T ≅ 0.05	0.21	0.41	0.71
1	Clean Conc. 8X20	99.87	Exp. Vm 90.85 Rock+T. 8.02	T < 0.03%	1.13	R+V = 1.07 T ≅ 0.06	0.15	0.35	.59
11	#1 Composite	99.85	Hand Picked: V = 89.15 R = 9.05 T = 1.66		0.15	R+V = 0.14 T < 0.01	1.67	1.87	2.17
12	#2 Composite	99.86	Hand Picked V = 84.85 R = 10.30 T = 4.71		0.14	R+V = 0.13 T < 0.01	4.72	4.92	5.22
5	#3 Composite	95.23	Exp. Vm 74.11 R+T 21.12	†T ≅ 0.21%	4.77	R+V = 4.58 T = 0.19	0.41	0.60	0.89

For +50M:
* Expand Vermiculite (by H_2O_2) exp.
** Rock + Tremolite — Seperated from Vermiculite by flotation
† Tremolite Content — detined by quant. X-ray method from the R+T sample fraction

a, assuming no tremolite in the expanded Vm

b assuming there is some tremolite but under the detection limit by X-rays (0.5%)

c. assuming max. Vm Content in Vm, which can not be detined accuratly by X-rays (0.5%)

2 Libby Ore Evaluation 20x65 Circuit & Fiver Composites (#4, #5)

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Sample #	Circuit	+70 M			-70 + 100 mesh		-100 mesh		Total Tremolite	
		Wt. (%)	Breakdown (%)	X-Rays of Obs.	Wt. (%)	Breakdown (%)	Wt. (%)	Breakdown (%)	min[a] (%)	max[b] (%)
9	Rough Feed 20x65	94.66	T. > 1%	high T.	4.42	R+V = 3.98 % T ≅ 0.44 %	0.92	R+V = 0.82 T ≅ 0.10	1.54	2.01
10	Clean Feed 20x65	91.77	T ≅ 0.9%	Int. low T	6.48	R+V = 5.51-5.53 % T = .65-.97 %	1.75	R+V = 1.67 T = .08	1.63-1.95	2.09-2.41
3	Rough Conc. 20x65	91.80	Exp. Vm = 57.38 % R+T = 34.42 % T < 0.34%	large white fiber balls	6.41	R+V = 6.35 % T < .06 %	1.79	Amorphous. no crystalline tremolite	< 0.4	< 0.86
4	Clean Conc. 20x65	92.65	Exp. Vm = 67.31 R+T = 25.34 T ≅ 0.25%	large white fiber balls	5.80	R+V = 5.34 % T ≅ 0.46 %	1.55	R+V = 1.52 T ≅ 0.03	0.74	1.20
6	#4 Composite	96.55	Exp. Vm = 72.86 R+T = 23.69 T ≅ 0.29%		2.77	R+V = 2.52 T ≅ 0.25 %	0.68	R+V = 0.65 T ≅ 0.03	0.52	1.00
13	#5 Composite	44.12	Exp. Vm = 22.93 R+T = 21.19 T ≅ 0.21%		35.69	R+V = 33.55 % T = 2.14 %	20.19	R+V = 19.09 T = 1.1	3.45	3.7

For +70M: * Vm were expanded chemically by H_2O_2
Rock + Tremolite were separated from chemically expanded Vm by flotation
Tremolite content was determined by quantitative x-ray methods of the R+T sample fraction

For -70+100M + -100M: Tremolite content was determined by quant x-ray method of the whole fraction

a. assuming no tremolite in expanded verm at all (0.2%)

b. assuming max. amount of tremolite in expanded Vm which can't be defined by x-rays accurately (0.5%)

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495668

CAMBRIDGE **CONFIDENTIAL** RECD APR 26 1977

TO: E. S. Wood

DATE: April 19, 1977

FROM: Julie C. Yang

SUBJECT: Tremolite Content in ZONOLITE® Products

CC: H. C. Duecker
H. A. Eschenbach
F. W. Eaton
W. R. Hanlon
R. M. Vining
B. R. Williams
J. W. Wolter
C. C. Ou
S. C. Vaughan
File: 71-046

ADMINISTRATIVE RECORD

OBJECTIVE:

The objective of this study is to determine the tremolite content in all ZONOLITE products made of both Libby and Kearney vermiculites. In a few cases, repetitious analyses were made for product used on job-sites, so that correlation can be made with the fiber counting results.

METHOD

When tremolite is determined from the product as received, in most products tremolite was not found by conventional analytical methods. The trace amount can be determined only when intensive concentration techniques are employed. Tremolite determinations are then made from the fractions by quantitative x-ray diffraction analysis and with the aid of petrographic microscopic examination.

1. Terra-Lite Vermiculites, Verxite, Redi-Earths and Metro-Mixes

The schematic method of analysis and the results have been reported in T&A 50110 with limited distribution. They are also reported here as shown in schemes 1, 2, and 3.

2. Scott Turf Builder

The method of concentration was very similar to that of Terra-Lite Vermiculite scheme #1, except in the water flotation step. A longer soaking period was needed to solubilize all the nutrients present, which was approximately 50% of the total weight.

3. ZIC, Attic Fill, Masonry Fill

Same concentration method as Terra-Lite (scheme #1).

EXHIBIT 4 Emergency No.

4. MONOKOTE

Analysis of tremolite in MONOKOTE was the most difficult and time-consuming procedure. The glass fibers were screened off, plaster of Paris was dissolved in water about 50-100 times the weight, expanded vermiculite was floated off, and all the washings were combined, filtered and dried. The filter paper and the organic matter were then burnt off; the remaining residue was x-rayed for the tremolite analysis. Detailed separation and concentration procedure is shown in scheme #4.

5. ZONOLITE 3300

Separation and concentration techniques are similar to that of MONOKOTE, but dilute acid (in HCl) was used to digest the portland cement binder instead of using large excess of water for solubilizing plaster of Paris. The procedure is shown in scheme #5.

RESULTS

A. Tremolite Content in ZONOLITE Products

Kearney

ID No.	Product Description	% Tremolite			
1	ZIC K-4 Kearney	5.466			
2	ZIC K-4/5 B	1.715			
4	Masonry Fill K-4	1.605			
9	Masonry Fill K-3	.0504			
11	MK-4 Kearney 3	<0.08			
13	MK-5 Kearney 3	<0.08			
17	Terra-Lite Kearney	4.319			
18	Terra-Lite T.R.	0.016			
20	Metro Mix 200 T.R.	(as rec'd) 0.398	(dried)* .477		
21	Redi-Earth T.R.	(as rec'd) 0.048	(dried) .071		
23 (5)	Vermiculite Carrier Grade #4, Kearney (St.Louis)	0.083	(<0.008)		
26	Metro-Mix 300, T.R.	(as rec'd) 0.081	(dried) 0.121		
27	Metro-Mix 350, T.R.	(as rec'd) 0.156	(dried) 0.259		

*Metro-Mixes and Redi-Earths were computed both in as-received basis and oven-dried basis since the product has substantial amount of moisture.

by

No.	Product Description	% Tremolite	
0	MK-4 (L-3) West Chicago	< 0.10	
6	Masonry Fill (L4D-18) West Chicago	0.01	
9	Terra-Lite, W. Chicago	0.035	
25	Attic Fill (L-2) W.Chicago	.013	
28	Redi-Earth (L) Santa Ana	(as rec'd) .031	(dried) .051
14	Redi-Earth (L) W. Chicago	<0.02	
15	Metro-Mix 200 (L) W. Chicago	(as rec'd)0.034	(dried)<.043
12	Zonolite 3300 (L-3) W. Chicago	<0.007	
3	Concrete Aggregate (L4D-18) W. Chicago	0.344	
16	Scott Turf Builder (L) Dark	<0.009	
22	Scott Turf Builder (L) Light	<0.009	

B. Tremolite Content in Zonolite Job-site Samples

ID No.	Product Description	Location	% Tremolite
8	ZK Roof Deck (K 4/5 B)	Montgomery, Ala.	2.828
9	Masonry Fill (K-3)	Columbus, Ohio	0.050
28	Redi-Earth (L-4)	Forest Service, Santa Ana	0.031 (.051)*
51	Monokote-5 (L-3)	San Diego	<0.106
54	Masonry Fill (K-4)	W.Palm Beach, Fla.	2.86
55	ZIC (K-4)	Edison H.S., Miami, Fla.	0.476
58	Masonry Fill (L-3) Mashburn	& Coe Bldg., Oklahoma	0.250
57	Monokote-4 (L-3)	Hyatt Regency, Dallas	0.240

*oven-dried basis

DISCUSSION and COMMENTS

1. Some of the Kearney products showed high "tremolite" content since x-ray diffraction method cannot distinguish massive tremolite (Hornblende?) and fibrous tremolite. Microscopically, most of the Kearney material showed trace or absence of fibers.

2. Tremolite fibers can be reduced if a screened vermiculite is used such as in vermite. We have observed that most of the fibers are concentrated in the fines.

To: E. S. Wood
From: J. C. Yang
April 20, 1977

3. The percentage of tremolite in several samples was expressed in less than a certain value which indicated that tremolite fiber was not detected by our x-ray method. The limit of detection for tremolite by x-ray diffraction technique is about 0.2%. When concentration factors were taken into consideration, the possible maximum tremolite content in each sample was indicated in the analyses.

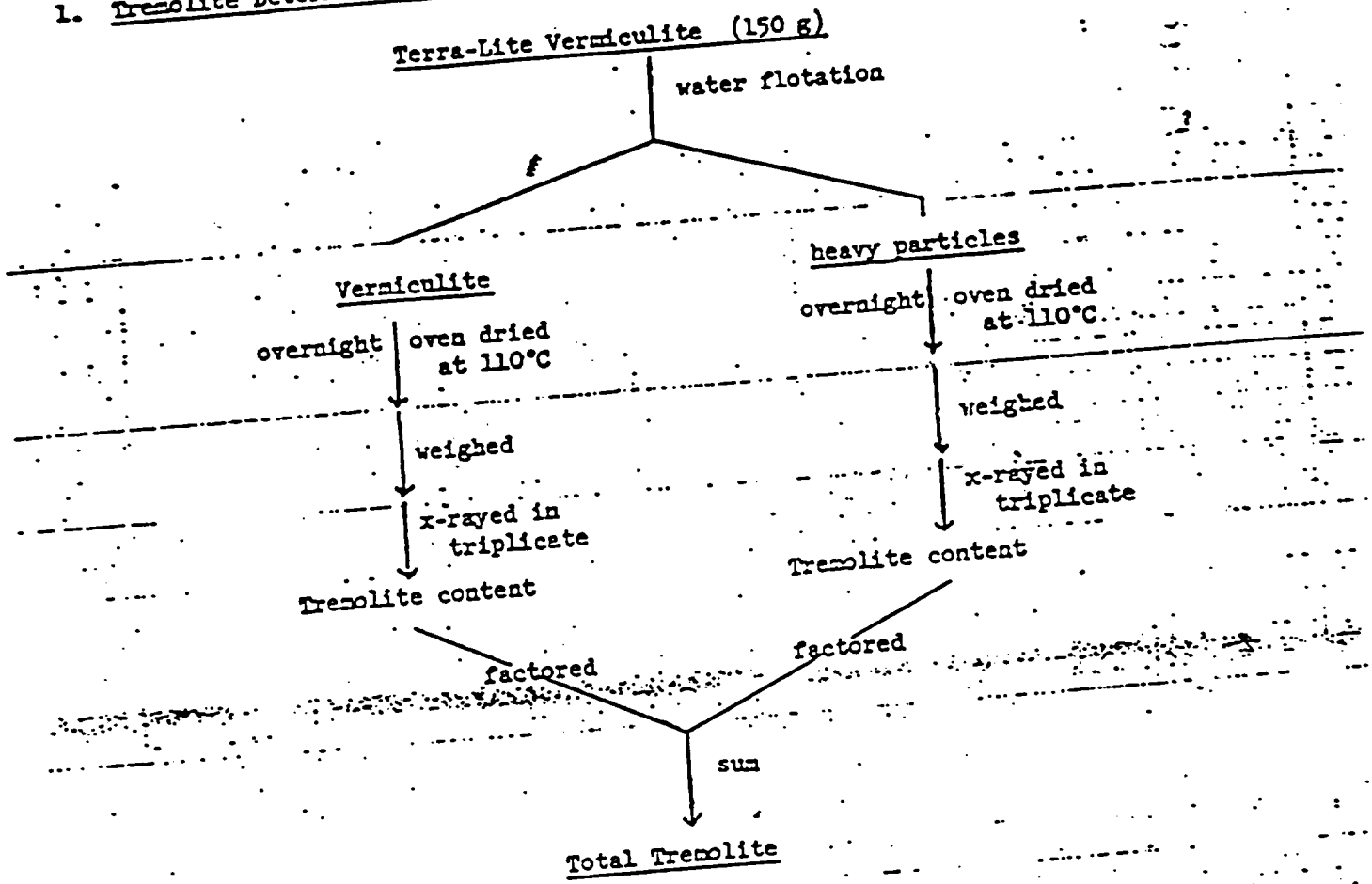
4. Most of the Monokote showed undetectable tremolite content except #57, an MK-4 product used at Hyatt Regency in Dallas, which showed a 0.2% tremolite; the value has been double checked and is real.

Julie C. Yang

JCY:clr

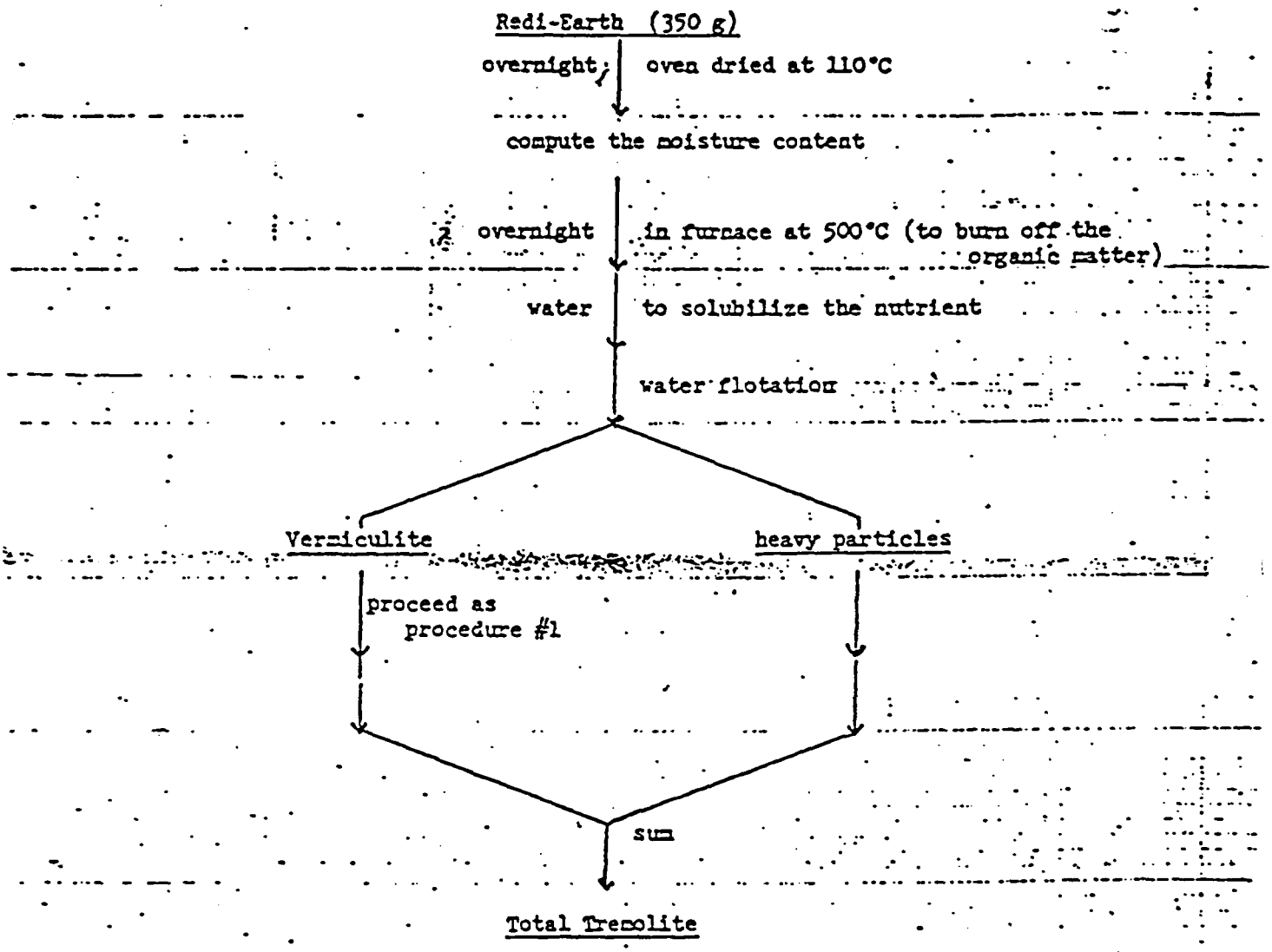
1. SCHEMATIC DIAGRAMS FOR TREMOLITE ANALYSIS

1. Tremolite Determinations in Terra-Lite Vermiculite



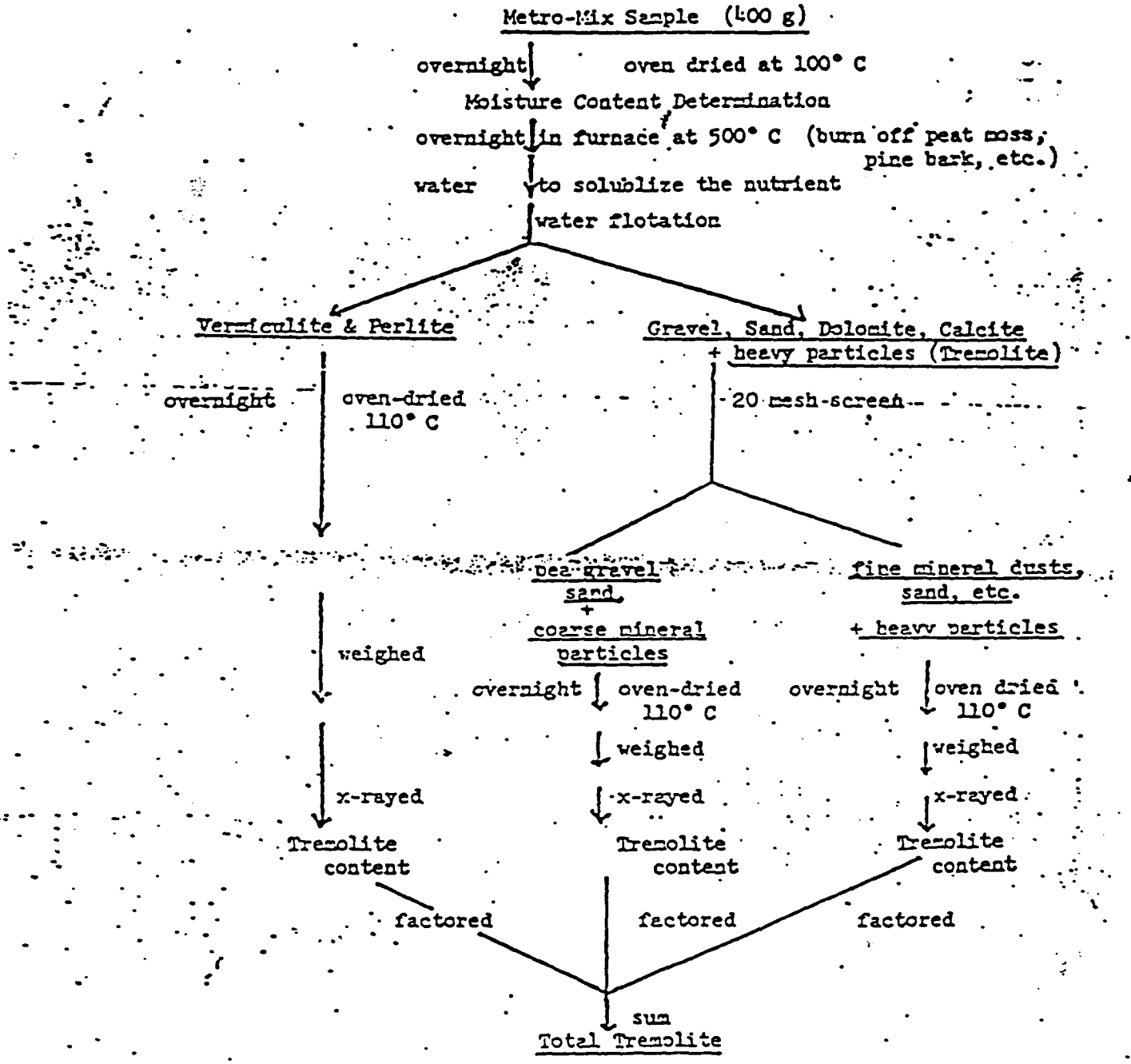
Julie C. Yang
April 19, 1977

2. Tremolite Determination in Redi-Earth



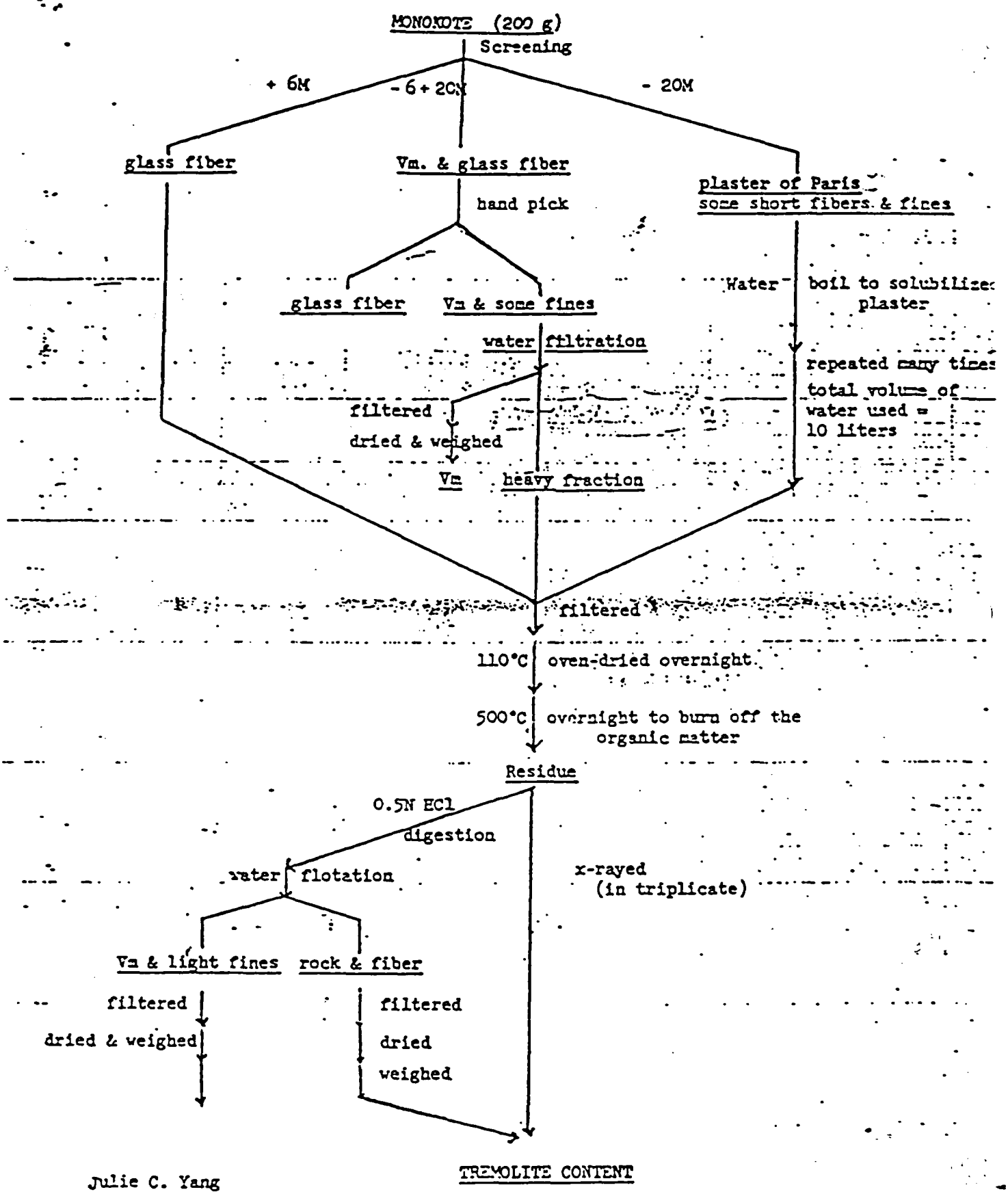
Julie C. Yang
April 19, 1977

3. Tremolite Determinations in Metro Mix



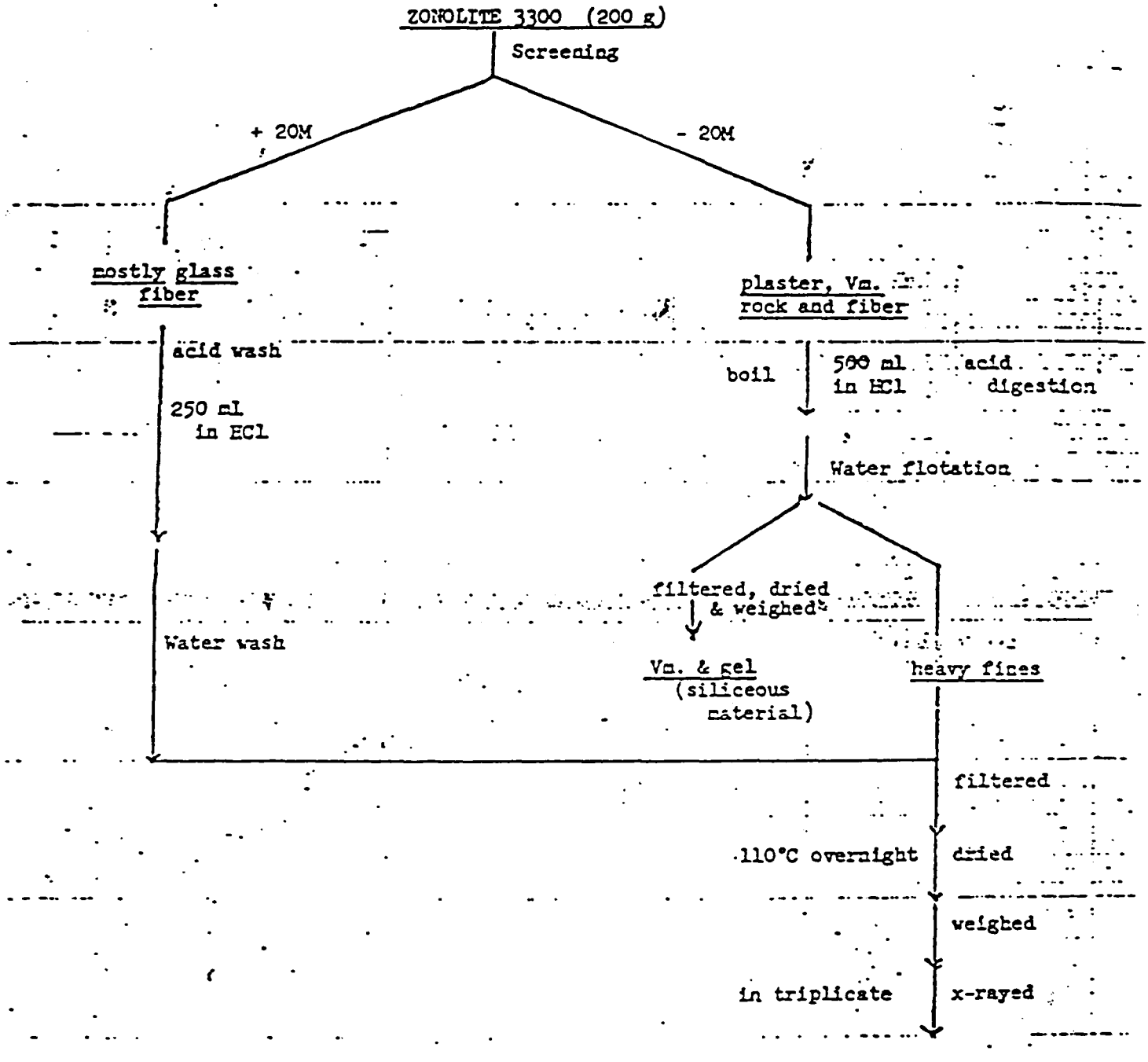
Julie C. Yang
April 19, 1977

4. TREMOLITE DETERMINATION IN MONOKOTE



Julie C. Yang
April 19, 1977

5. TREMOLITE DETERMINATION IN ZONOLITE 3300



Julie C. Yang
April 19, 1977

495669

CONFIDENTIAL

RECD MAY 25 1977

CAMBRIDGE

02225285

TO: E. S. Wood

DATE: May 16, 1977

FROM: J. C. Yang

SUBJECT: Tremolite & Vermiculite Content in Libby & Kearney Ore Deposits and Expanded Vermiculites

CC: R. M. Vining, B. R. Williams, J. W. Wolter, W. R. Hanlon, D. M. Kirven, R. C. Ericson, R. H. Locke, H. C. Duecker, H. A. Eschenbach, B. A. Blessington, C. C. Ou, F. W. Eaton, O. M. Favorito, S. C. Vaughan
File: 71-048/049

OBJECTIVE

The objective of this study is to determine the vermiculite and tremolite content in ore concentrate and expanded vermiculite from the Libby and Kearney mills. A sample of the head feed from the Libby mills, from which all the Libby ore samples were derived, is also analyzed as a check for the effectiveness in fiber removal of the Libby operation.

The samples analyzed below are single samples of concentrate or expanded product, selected at random. We do not know how accurately these samples represent the average with respect to tremolite (or amphibole mineral) content. Further sampling will be required to better establish more typical or average values.

The reported tremolite content may include other amphibole minerals, particularly hornblende, which cannot be distinguished from tremolite.

SAMPLE DESCRIPTION

All the analyses made in this report were single sample analyses. From the materials submitted in 5-10 lb. quantities, they were quartered very carefully and repeatedly until the desired sample sizes (200-300 grams) were obtained, which were expected to be fairly representative. However, the range of variations in field sampling and in the geological formations were not established, so that the results observed may only indicate a ballpark figure with ±10% of accuracy.

02225286

To: E.S.Wood
From: J.C.Yung
Date: May 16, 1977

Tremolite & Vermiculite Content in Libby & Kearney Ore Deposits and Expanded Vermiculites

ID No.	Description	Date and Source
99952-31	Ore Concentrate L-1	3/10/77 - R. L. Oliverio
99952-32	Ore Concentrate L-2	
99952-33	Ore Concentrate L-3	
99952-34	Ore Concentrate L-4	
99952-35	Ore Concentrate L-5	3/1/77 - E. D. Lovick
99952-36	Ore Concentrate K-3	3/7/77 - O. F. Stewart
99952-37	Ore Concentrate K-4	
99952-38	Ore Concentrate. K-5	
99952-39	Expanded Vermiculite L-1	3/21/77 - F. W. Eaton
99952-40	Expanded Vermiculite L-2	
99952-48	Expanded Vermiculite L-3 (Terra-Lite)	3/9/77 - F. W. Eaton
99952-41	Expanded Vermiculite K-3	3/3/77 - O. F. Stewart
99952-42	Expanded Vermiculite K-4	
99952-43	Expanded Vermiculite K-5	
99952-46	Libby Head Feed - a composite of 3 shifts	3/9/77 - R. L. Oliverio

METHOD

1. Tremolite Analysis of Libby #1 and #2 Concentrate:

Since the fiber bundles and the rock aggregates are unusually large, tremolite fiber bundles and rocks were first separated by hand-picking of a carefully quartered sample. The vermiculite was then separated from the rock by screening. Rocks and fines in the -50 mesh fraction were x-rayed for quantitative determination of tremolite. The total tremolite was obtained as the sum of factored portions from hand-picked and the fine portions. The scheme of analysis is shown in Figure 1.

2. Tremolite Analysis of #3 Ore Concentrate:

The concentration of rock fines and tremolite fiber fractions are shown in Figure 2. Vermiculite was separated by chemical exfoliation with 30% H_2O_2, followed by water flotation.

15025894

To: E.S.Wood
From: J.C.Yang
Date: May 16, 1977

Tremolite & Vermiculite Content in Libby & Kearney Ore Deposit.. and Expanded Vermiculites

02225287

3. Tremolite Analysis of #4 and #5 Ore Concentrate:

A set of finer screens than the ones used in analyzing #3 ore was selected for this separation. A diagram of the procedure is shown in Figure 3.

4. Tremolite Analysis of Expanded Vermiculite (size #2 to #5)

The expanded vermiculites are easier to work with since they were expanded already and the percentage of rocks and fines were lower than those of the corresponding ore concentrates.

The procedure for analyses is shown in Figures 4 and 5, respectively.

5. Tremolite Analysis of the Head Feed:

The head feed sample from the Libby mill was obtained one day before the ore composite collection from the screening plant, and is the starting material from which the ore composites were obtained. The analysis was more complicated than the others since the size varied over a wide range and the non-vermiculite portion was very high. The tremolite concentration procedure is shown in Figure 6.

6. Vermiculite Analysis of Ore Concentrates:

To cross-check the vermiculite analysis from the scheme shown in Figures 2 and 3, for ore composites #3 and #5, a 100 g ore sample was taken and expanded in a furnace for 5 minutes at 1500°F., then allowed to cool at ambient conditions for half an hour. In general, a weight loss of about 7% resulted from heat expansion. By previous experience, a higher vermiculite yield will result from chemical expansion since some of the poorly weathered vermiculite will not readily respond to heat expansion but will expand in H_2O_2. The complete analyses of the Libby and Kearney vermiculites are shown in Tables 1 and 2.

7. Evaluation of Fine Fiber Content:

In Table 1, a breakdown of the tremolite fiber in ore concentrate by size fraction is also shown. The fines (-50M for size L-1 to L-3; -100M for size L-4 and L-5) can be considered to be the maximum limit of the respirable fiber portion (provided no further vigorous mechanical degradation of the material takes place in handling).

We have also hand-picked the fiber bundles from two L-2 ore concentrate samples and run them through the air-elutriation column built in the laboratory. We then collected the airborne particulate through a series of screens and then on a wet filter under vacuum. The screens used were graded to eliminate the blockage of the filter by large dust aggregates and long fibers. This experiment indicated the fiber bundles were fairly stable. At the end of 30 minutes of the air elutriation, the results are as follows:

15025895

To: E.S.Wood
From: J.C.Yang
Date: May 16, 1977

Tremolite & Vermiculite Content
in Libby & Kearney Ore Deposits
and Expanded Vermiculite

02225289

3. The fine size (and potentially respirable size) tremolite fiber contents in the Libby ore composites were very low (in the order of 0.01%) for L-2 ore concentrate. Fiber bundles usually remain intact under normal operations and are concentrated in the stoner. Some of the small fibers present between vermiculite plates may be loosened during the expanding operation, the amount yet to be determined. Another possible source of respirable size fibers in expanded product is the breakdown of fiber bundles during heat expansion. This will be investigated shortly. When all the sources are identified and the approximate amounts become known, a method for more effective removal or reduction can be sought with some confidence.

J.C. Yang

Julie C. Yang

JCY:mlr

attachments

15025897

To: E.S.Wood
From: J.C.Yang
Date: May 16, 1977

Tremolite & Vermiculite Content in Libby & Kearney Ore Deposits and Expanded Vermiculite

02225290

TABLE 1

Tremolite Content of Ore Concentrate

ID No.	Description	Date	% Vm.	% Tremolite		% Total Tremolite*
-31	L-1	3/10/77	91.7	(+50M) (-50M)	1.2 .005	1.2
-32	L-2	3/10/77	91.2	(+50M) (-50M)	2.5 .018	2.5
-33	L-3	3/10/77	78.1	(+50M) (-50M)	.653 .013	0.7
-34	L-4	3/1/77	70.1	(+70M) (-70 +100M) (-100M)	1.495 .232 .009	1.7
-35	L-5	3/1/77	63.9	(+70M) (-70 +100M)	.119 1.016 1.913	3.0
-36	K-3	3/1/77	72.0	(+50M) (-50M)	1.60 .158	1.8
-37	K-4	3/1/77	75.1	(+70M) (-70 +100M) (-100M)	8.903 .554 .492	10.0
-38	K-5	3/1/77	76.6	(+70M) (-70 +100M) (-100M)	0.874 2.070 13.034	15.9
-46	Head Feed, Libby	3/9/77	7.0**	(+6M) (-6 +20M) (-20 +70M) (-70M)	1.302 .684 1.235 .609	3.8

*Includes all amphibole minerals.

**The material floated after expanded with 30% H_2O_2.

CONFIDENTIAL

15025898

To: E.S.Wood
From: J.C.Yang
Date: May 16, 1977

Tremolite & Vermiculite Content in Libby & Kearney Ore Deposits and Expanded Vermiculite

02225291

TABLE 2

Tremolite Content of Exponded Vermiculite

ID No.	Description	Date Collected	% Vermiculite	% Tremolite
99952-39	L-1	3/18/77	97.7	0.074
99952-40	L-2	3/18/77	97.1	0.028
99952-48	L-3	3/9/77	97.7	0.049
99952-41	K-3	3/3/77	91.0	1.6*
99952-42	K-4	3/3/77	79.4	7.9*
99952-43	K-5	3/3/77	48.7	interference

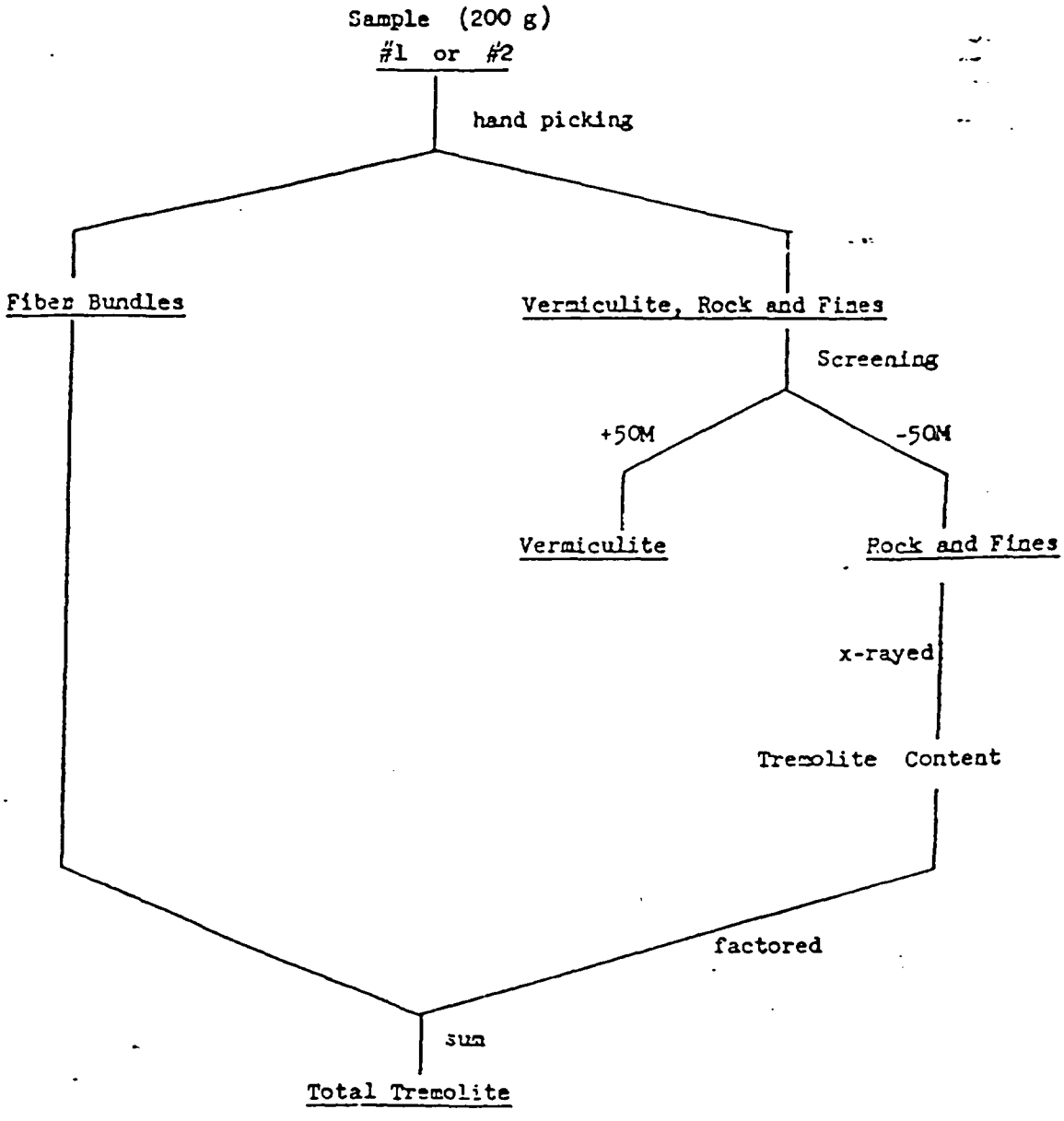
*Includes all amphibole minerals.

CONFIDENTIAL

15025899

FIGURE 1

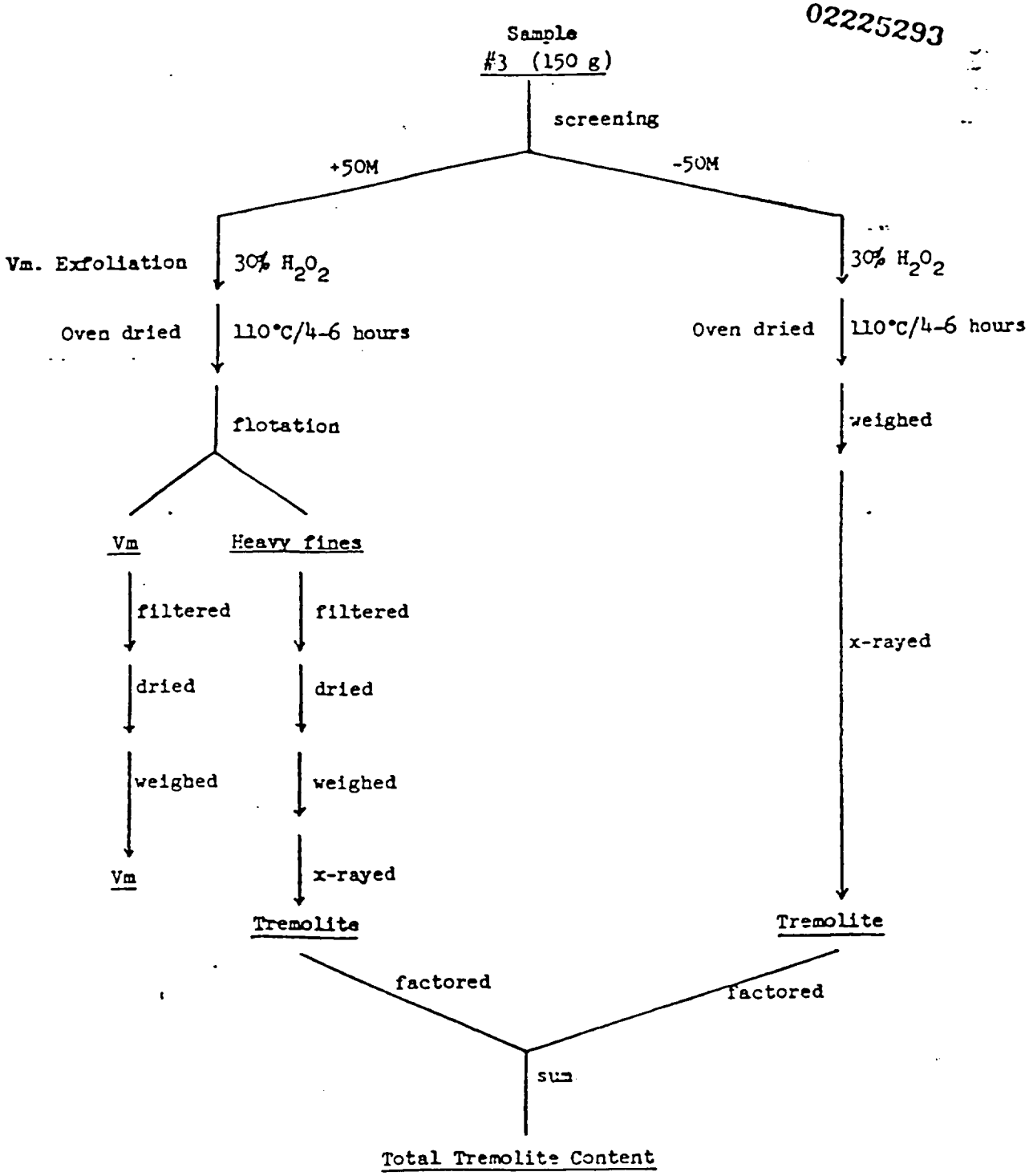
1) TREMOLITE ANALYSIS OF #1 and #2 ORE COMPOSITES



J.C.Yang:mlr
5/16/71

FIGURE 2

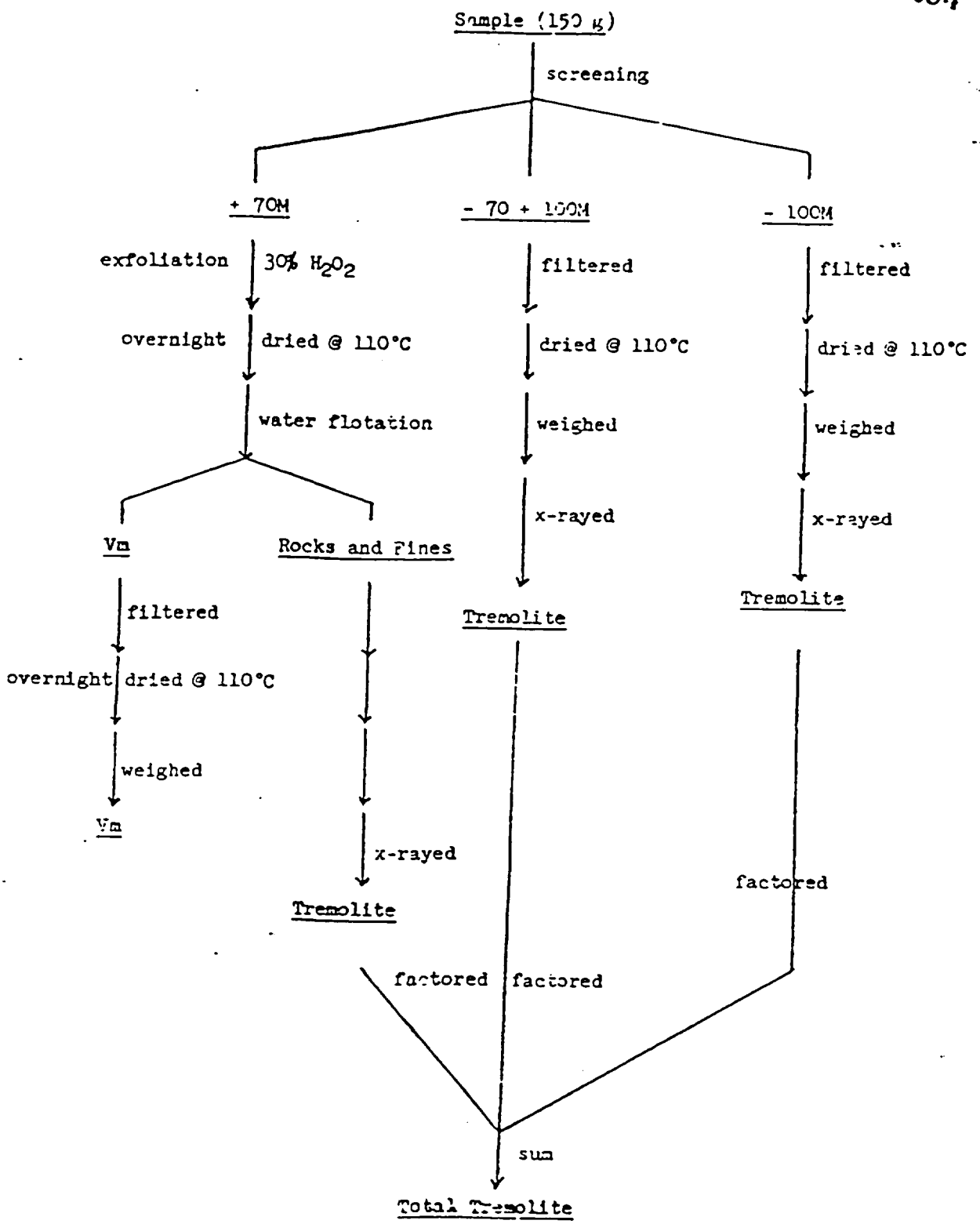
2) TREMOLITE ANALYSIS OF #3 ORE COMPOSITE



5/16/77

FIGURE 1

3) TREMOLITE ANALYSIS OF #4 and #5 ORE COMPOSITE



J.C.Yang:mlr
5/16/77

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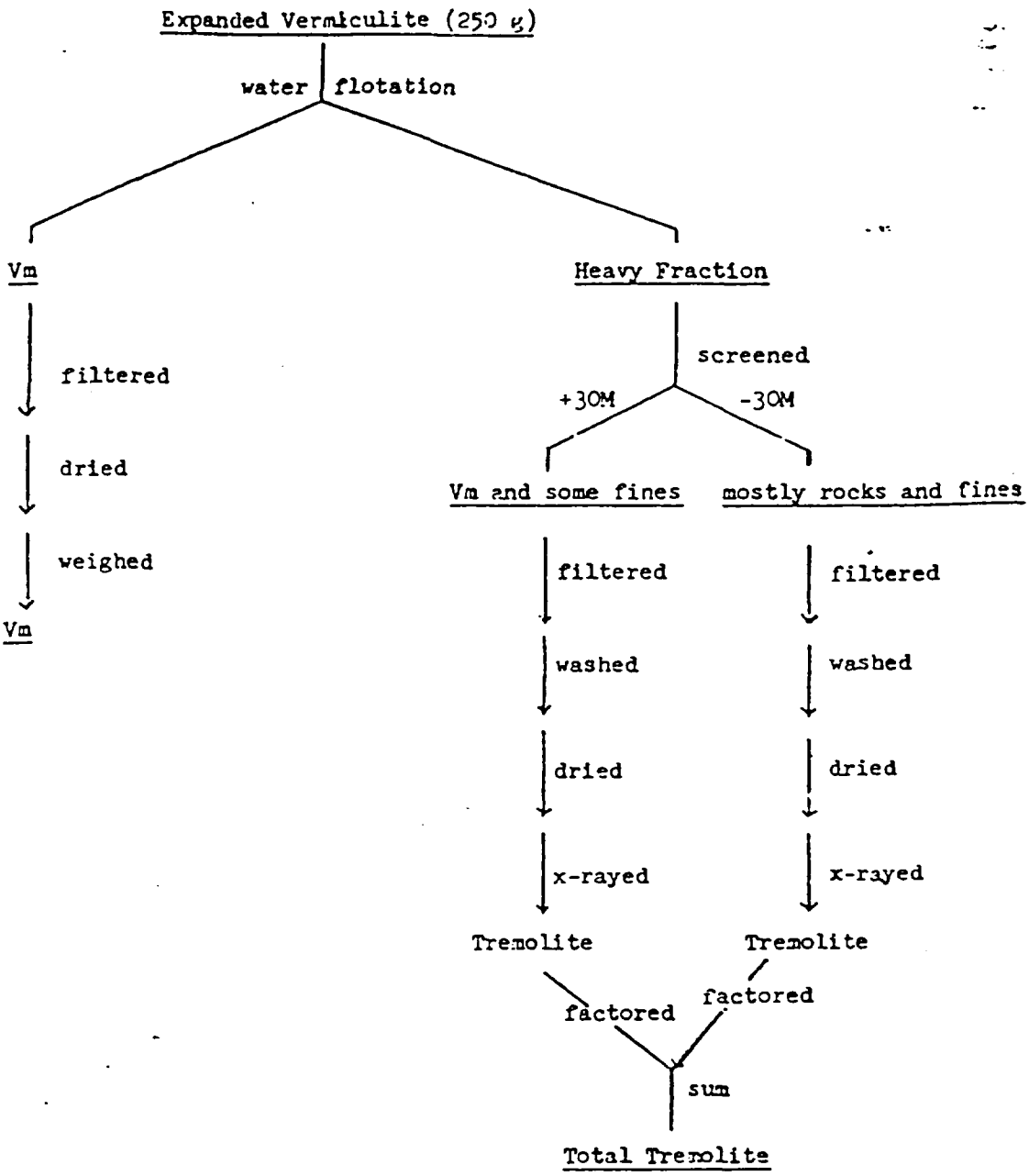
FIGURE 4

02225295

4) TREMOLITE ANALYSIS OF #2, #3 AND #4 EXPANDED VERMICULITE



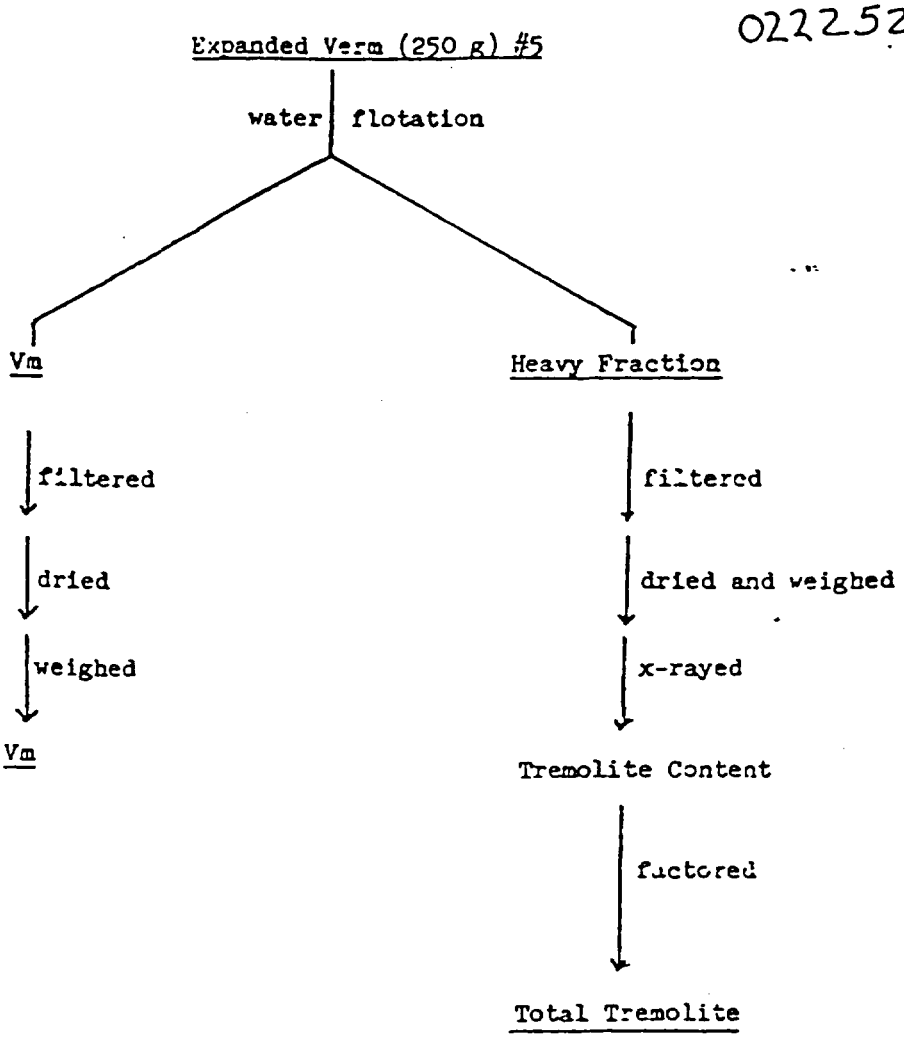
J.C.Yang:mlr
5/16/77

15025903

FIGURE 5

5) TREMOLITE ANALYSIS OF #5 EXPANDED VERMICULITE

02225296

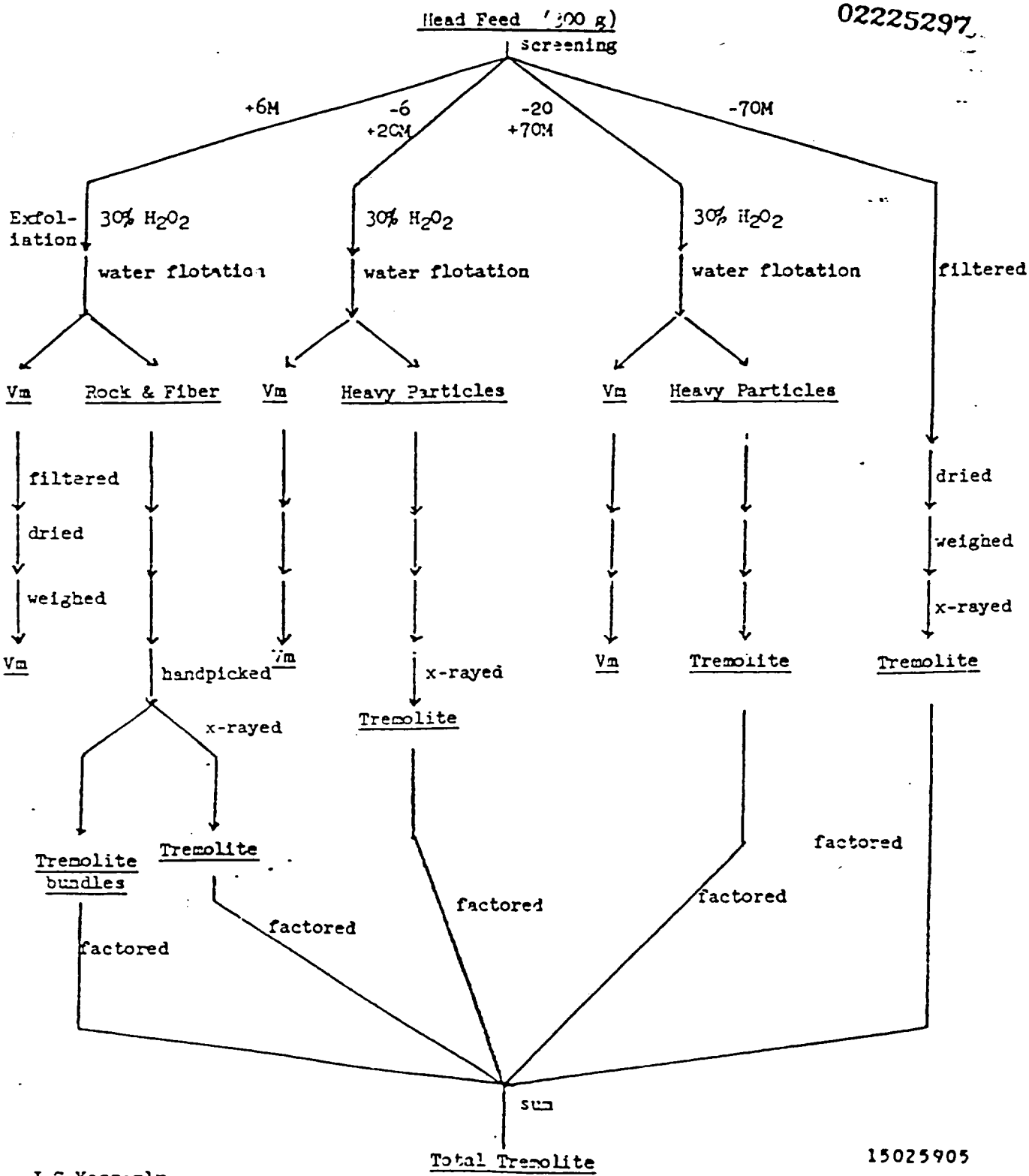


J.C.Yang:mlr
5/16/77

15025904

6) TREMOLITE ANALYSIS OF THE HEAD FEED

A head feed from Libby was analyzed from which the ore composite L-1, L-2, L-3 and L-4 were obtained.



J.C.Yang:nlr
5/16/77

CONSTRUCTION PRODUCTS DIVISION

REQUEST FOR TECHNICAL SERVICE

PAGE 1

03645C89

NUMBER: 67659

GROUP: CPD Zonolite

DATE: February 21, 1978

CHARGE NO.: 984

REQUESTOR: F. W. Eaton

MARKETING or MANUFACTURING APPROVAL:

NAME: F. W. Eaton

APPROVED:

PROBLEM TITLE: Determine % tremolite in CPD vermiculite and/or users product.

SIGNIFICANCE: Correlation between user personnel exposure to tremolite at job site and % tremolite in vermiculite used and/or product produced.

SPECIFIC OBJECTIVE: Collect data on tremolite content on vermiculite products used by CPD customers.

495670

ADMINISTRATIVE RECORD

SUGGESTED APPROACH:

DEADLINE (Last day information will be of value): Routine-reference user exposure fiber analysis T & A.

DETAILS OF PROBLEM:

1. L #2 W. Chicago material used at American Hospital Supply/Waukegan.
2. L #1 Attic Milwaukee material used at Aldrich Chemical/Milwaukee.

Above used as packaging material.

ACCEPTED BY RESEARCH DEPT.: DATE: 2/21/78

ASSIGNED TO: S. Vaughan

ADDITIONAL COPIES: Original to Library, H.C. Duecker, F.W.Eaton, T.E.Hamilton, J.W.Wolter, H.S.Wood, CPD-TSA, File: 984

CONFIDENTIAL

15035521

REQUEST FOR TECHNICAL SERVICE

NUMBER: 67659
GROUP: CPD ZONOLITE
ACTUAL COST: $150.00
REPORTING DATE: March 1, 1978

03645090

SUMMARY:

Two samples submitted were analyzed for their tremolite content. The results are shown below.

RESULTS:

Sample ID	Description	User	% Tremolite
295-3-1	L-2, Chicago material	American Hospital Supply in Waukegan, Illinois	0.035
295-3-2	L-1, Attic Milwaukee material used as packing material	Aldrich Chemical, Milwaukee, Wisconsin	0.083

Reference: Notebook 295-3 (SV)
X-ray chart: 900 #12

Julie C. Yang

JCY:mlr

15035522

2

495699

CONFIDENTIAL

CAMBRIDGE

TO: H. C. Duecker

DATE: February 23, 1976

FROM: Julie C. Yang

SUBJECT: Libby Ore Evaluation - Ore Impurities

CC: H. A. Brown
J. W. Wolter
R. L. Oliverio/Libby
R. J. Kujawa/Libby
G. G. Vaplon/Libby
O. F. Stewart/Enoree
R. H. Locke
J. L. Young
File: 71-048

03627800

PURPOSE

The objective of this investigation is to determine the tremolite content for each of the three mill circuits and end products at Libby.

SAMPLE SELECTION

Samples have been collected by G. Vaplon:

(a) material which entered the circuit,
(b) material which came out of the circuit,
(c) screened plant products as control and comparison with (a) & (b).

Fourteen materials were received:

(1)	Clean Conc.	8 x 20	
(2)	Rough Conc.	8 x 20	
(3)	Rough Conc.	20 x 65	
(4)	Clean Conc.	20 x 65	
(7)	Rough Feed	8 x 20	
(8)	Clean Feed	8 x 20	
(9)	Rough Feed	20 x 65	
(10)	Clean Feed	20 x 65	
(11)	#1 Composite		
(12)	#2 Composite		
(5)	#3 Composite		
(6)	#4 Composite		
(13)	#5 Composite		
(14)	Humphrey Sizer Concrete	12/3/75	9:00 a.m.

EXPERIMENTAL

I) Humphrey Sizer

1. Separation

The rock and fiber were separated from the vermiculite plates by hand-picking.

2. Method of Analysis

Each portion has been weighed carefully and then x-rayed for their mineral content.

20152820

068ETX02076

To: H. C. Duecker
From: Julie C. Yang
Feb. 23, 1976

Libby Ore Evaluation - Ore Impurities

03627801

3. Results and Accuracy

	Wt. %	Accuracy %	
Vermiculite	86.71	86.71 ± 0.43	approx.*
Rock	10.58	10.58 ± 0.05	approx.*
Tremolite	2.71	2.71 ± 0.01	
Total	100.00		

*The rock content may be higher than the figure shown at the expense of vermiculite, since some of the granules can be classified as vermiculite fine aggregates (showed vermiculite x-ray pattern) but may not be expandable as we previously found (report 11/3/75 - Properties of Libby Vermiculite Ore). The fiber portion showed a good x-ray pattern of pure tremolite with no rock contaminations.

II) 8 x 20 Circuit and End Products #1, 2 & 3

1. Separation

The samples in this group were sized by Ro-Tap screening to +50 and -50 fractions. 100 gram vermiculite sample was Ro-Tapped for 16 minutes total, a ten minute increment first, then three 2-min.consecutive intervals to insure the achievement of constant weights.

Then from the +50 size fractions, fibers were hand-picked and weighed. The bulk materials remaining were then chemically expanded with 30% H_2O_2 individually. The light expanded vermiculite thus was separated from the heavy rocks and fiber bundles by water flotation. Both portions were collected, dried and weighed, then ground to -100 mesh and subjected to x-ray examination.

2. Method of Analysis

Tremolite remaining in the samples was determined by quantitative x-ray diffraction analysis, and the values were added to those of the hand-picked tremolite. In quantitative x-ray analysis a calibration curve was constructed to determine tremolite by adding a known amount of Libby tremolite (hand-picked from #2 composite, opened and cleaned) to a hand-picked pure vermiculite sample. The curve was made for determinations up to 10% tremolite.

The total area under the $2\theta = 28.5°$ in the diffraction pattern, the peak responded to the max intensity peak of tremolite, was computed for the quantitative studies, and a second peak (height only at $2\theta = 10.5°$) was employed as a check for the interference (Figure 1).

20152821

03627802

3. Result and Accuracy of Analysis

Experimental results are listed in Table 1. Since the detection limit of tremolite by x-rays is about 0.2% in a specific sample, for very low concentration occurence tremolite has to be concentrated in the sample by removing the bulk of vermiculite. Vermiculite can be removed easily by chemical expansion with 30% H_2O_2 followed by flotation.

On the chart, three tremolite contents (actually the range) were given based on the detection limitations.

4. Comparison of Material from 8 x 20 Circuit and End Products Composite #1, #2 and #3

The rock content of composites #1 and #2 are in line with those of the concentrates in the 8 x 20 circuit, but the tremolite content in these composites are definitely higher than the concentrates. The exceptionally high tremolite content is noted in Composite #2. The fiber contents in the 8 x 20 concentrates are slightly less than those in the corresponding feeds.

III) 20 x 65 Circuit and End Products Composites #4 and #5

1. Separation and Analysis

The samples in this group were sized by Ro-Tap screening to 3 fractions, namely +70, -70 +100 and -100 mesh size using the procedure described in Section II 1.

In the +70 fraction of rough and clean concentrates, the fine fibers present were balled up to pea-sized white balls, which were separated by gentle screening. The fiber balls were retained on a 50 mesh screen and weighed. To check the fiber content, the weighed fiber balls were broken and redistributed in the sample and subjected to quantitative x-ray determination. Since the fiber contents were very low, vermiculite in these samples were expanded chemically and then removed by flotation prior to x-ray analysis.

In the -70 +100 and -100 mesh fractions, tremolite was determined directly from the sample as received; since the vermiculites present in these sizes are fairly small, the expansion and flotation will not separate the material effectively.

20152822

To: H. C. Duecker
From: Julie C. Yang
Feb. 23, 1976

Libby Ore Evaluation - Ore Impurities

2. Results and Accuracy

The experimental data is presented in Table 2. In the +70 fraction of the rough and clean concentrates, the white fiber balls of tremolite separated by gentle screening were found to be 0.32 and 0.22% respectively, in comparison to the values of <0.34 and 0.25% by x-ray method.

Again the tremolite contents were given in a range (max. and min.) in Table 2, based on detection limitations of the method.

3. Comparison of Material for 20 x 65 Circuit and End Products #4 and #5

The fiber contents in the concentrates of the 20 x 65 circuit are definitely less than those in the corresponding feeds, and also in line with the end product #4 composite.

End product #5 showed quite a high fiber content (~3.5%) and also a high rock content. For a rough estimate, the unexpanded material in this composite is close to 40% of the total.

OBSERVATIONS and COMMENTS

1. In the 8 x 20 circuit and the end product #1 and #2, most of the fibers present are in heavy bundles and very small amounts of fine fibers except some adhered to the surface of the vermiculite platlets.

2. In the 20 x 65 circuit, most of the fibers present are opened fibrils or smaller bundles. They tend to ball up into small white spheres while the sample is being sized by screening.

3. In the end products #4 and #5, the fibers are too short to form balls but are distributed widely throughout the matrix.

4. From Tables 1 and 2, the concentrates in both circuits showed relatively less fiber than in the feeds.

5. The expansion of vermiculite followed by flotation is a good method for separating the vermiculite from the rocks and the fiber, and the fiber content is then determined by x-rays but the method is good only when the vermiculite size is reasonably large (~ 70 mesh or larger).

6. For the small sized vermiculite samples, the tremolite content can be determined only from the sample directly by x-rays quantitatively. If the need ever came to determine the rock content in the vermiculite, chemical delamination method with 15% LiCl can be employed. The method has been described in a previous report (T&A 48522, 9/12/75).

To: H. C. Duecker
From: Julie C. Yang
Feb. 23, 1976

Libby Ore Evaluation - Ore Impurities

03827S01

CONCLUSIONS

1. The possible tremolite content of end products of each size and of concentrates from the three circuits are:

Circuit	Tremolite Contents, percent Range	Mean
Humphrey Sizer	2.70 - 2.72	2.71
8 x 20		
Rough concentrate	0.21 - 0.71	0.46
Clean concentrate	0.10 - 0.59	0.35
20 x 65		
Rough concentrate	0.4 - 0.86	0.63
Clean concentrate	0.74 - 1.20	0.97
End Product		
Composites #1	1.67 - 2.17	1.92
#2	4.72 - 5.22	4.97
#3	0.41 - 0.89	0.65
#4	0.52 - 1.00	0.76
#5	3.45 - 3.97	3.71

2. Based on the experimental data, the approximate amount of tremolite present in tons per day, out of each of the three circuits, will be as follows:

Circuit	Total Materials out of the circuit (tons/day)*	Mean Tremolite Content (tons/day)
Humphrey Sizer	220	5.96
8 x 20	295	1.16
20 x 65	513	4.10

*based on 22 hours in a day.

3. The #2 composite showed the highest tremolite content (even more so than #5), and the fibers present are mostly in heavy bundle form, visible to the eye. This fact is also true for the material in the 8 x 20 circuit and other coarse end products #1 and #3. The tendency of fiber balling in the 20 x 65 circuit shows that the fibers are more opened or in thinner bundles in addition to some extra fines distributed throughout the end products #4 and #5, which will lead to the belief that there is some degree of down screening.

20152824

To: H. C. Duecker
From: Julie C. Yang
Feb. 23, 1976

Libby Ore Evaluation -
Ore Impurities

03627805

4. For quantitative x-ray analysis, the detection limit is about 0.2%. Because of the variation in x-ray response from sample to sample (variables such as orientation, sample thickness, and packing conditions), the accuracy of these determinations is approximately ±0.5% of the tremolite present. Therefore, in Tables 1 and 2, computations showed the maximum and minimum tremolite content possible to be present in the sample.

5. A previous report on Libby vermiculite and tremolite density determinations (1/13/76) showed an appreciable density difference between tremolite (2.92 - 3.1) and vermiculite (2.28 - 2.61 depends on the degree weathering) and the difference in morphology. Tremolite can be separated from vermiculite by air elutriation technique based on the difference in velocity of particle settling. Meanwhile, the vermiculite plates can be "polished" by removing some of the fine dust and fiber adhered to the surface in the air stream. A separate report will be written to describe the details of that aspect shortly.

6. The conclusions reached assume the samples are all representative samples of the operation. In reality, we know we have considerable variation in feed quality from minute to minute, hour-to-hour, and certainly from pile-to-pile. This experiment should be repeated to obtain a better feel for this variation. The sampling technique is probably the most significant problem in the study. Reasonably good analytical results can be obtained although very time-consuming. About $2.0M of laboratory time will be required to repeat this test.

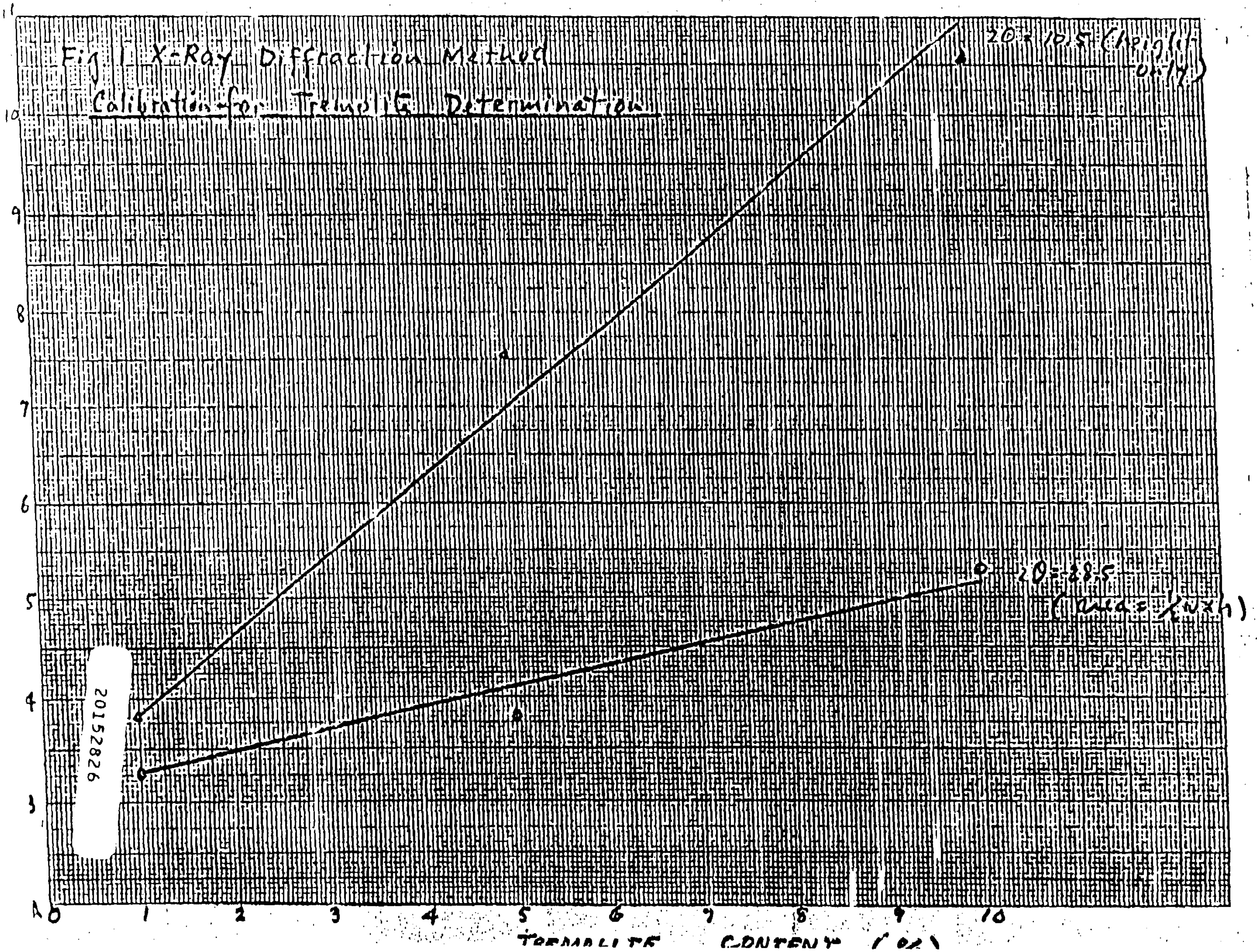
Julie C. Yang

JCY:mlr

Attachments

20152825

03627806



20152826

TABLE 1

Libby Ore Evaluation – 8X20 Circuit & Coarse Composites (# 1, # 2 + # 3)

20132827

03627807

Sample No.	Circuit	+50 mesh Wt (%)	+50 mesh Breakdown (%) Obs.	-50 mesh wt. (%)	-50 mesh Breakdown (%)	Total Tremolite Content min (%)	Tremolite Content Int.[b] (%)	Tremolite Content max (%)[c]
7	Rough Feed 8X20	99.88	Very low T <0.5%	0.12	Det'd by x-ray: V = 0.05, T = 0.07	0.5	0.7	1.0
8	Clean Feed 8X20	99.53	Low T <1%	0.47	R+V = 0.39, T = 0.08	1.0	1.2	1.5
2	Rough Conc. 8X20	99.05	Exp. Verm. 83.21; Rock+Trem** 15.84 {T = 0.16}	0.95	R+V = 0.90, T ≅ 0.05	0.21	0.41	0.71
1	Clean Conc. 8X20	99.87	Exp. Vm 90.85; Rock+T. 8.02 {T<0.08}	1.13	R+V = 1.07, T ≅ 0.06	0.15	0.35	.59
11	# 1 Composite	99.85	Hand Picked: V = 89.15, R = 9.05, T = 1.66	0.15	R+V = 0.14, T < 0.01	1.67	1.87	2.17
12	# 2 Composite	99.86	Hand Picked: V = 84.85, R = 10.30, T = 4.71	0.14	R+V = 0.13, T < 0.01	4.72	4.92	5.22
5	# 3 Composite	95.23	Exp. Vm 74.11; R+T 21.12 {T ≅ 0.21}	4.77	R+V = 4.58, T = 0.19	0.41	0.60	0.89

For +50M: * Expand Vermiculite (by H_2O_2) exp. Vermiculite; ** Rock + Tremolite – Separated from Vermiculite by flotation; † Tremolite Content – detied by quant. x-ray method from the R+T sample fraction [illegible] the whole sample

a, assuming no tremolite in the expanded

b. assuming there is som tremolite but under the detectg limit by x-rays (

c. assuming max Vm Content in Vm, which can not be detined accura by x-rays (0.5%)

2 Libby Ore Evaluation — 20x65 Circuit & Finer Composites (#4, #5)

20152828

03627808

Sample	Circuit	+70M Wt. (%)	+70M Breakdown (%)	+70M X-Rays vs Obs.	-70+100 mesh Wt. (%)	-70+100 mesh Breakdown (%)	-100 mesh Wt. (%)	-100 mesh Breakdown (%)	Total Tremolite min[a] (%)	Total Tremolite max[b] (%)
9	Rough Feed 20x65	94.66	T. > 1%	high T.	4.42	R+V = 3.98 % T ≅ 0.44 %	0.92	R+V = 0.82 T ≅ 0.10	1.54	2.01
10	Clean Feed 20x65	91.77	T ≅ 0.9%	Int. low T.	6.48	R+V = 5.51(-5.53 % T = .65-.97 %	1.75	R+V = 1.67 T = .08	1.63-1.95	2.09-2
3	Rough Conc. 20x65	91.80	Exp. Vm = 57.38 % R+T = 34.42 %	large white fiber balls T ≤ 0.34%	6.41	R+V = 6.35 % T < .06 %	1.79	Amorphous, no crystalline tremolite	<0.4	<0.86
4	Clean Conc. 20x65	92.65	Exp. Vm = 67.31 R+T = 25.34	large white fiber balls T ≅ 0.25%	5.80	R+V = 5.34 % T ≅ 0.46 %	1.55	R+V = 1.52 T ≅ -0.03	0.74	1.20
6	#4 Composite	96.55	Exp. Vm = 72.86 R+T = 23.69	T ≅ 0.29%	2.77	R+V = 2.52 T ≅ 0.25 %	0.68	R+V = 0.65 T ≅ 0.03	0.52	1.00
13	#5 Composite	44.12	Exp. Vm = 22.93 R+T = 21.19	T ≅ 0.21%	35.69	R+V = 33.55 % T = 2.14 %	20.19	R+V = 19.09 T = 1.1	3.45	3.7

For +70M: * Vm were expanded chemically by H_2O_2. Rock + Tremolite were separated from chemically expanded Vm by flotation. Tremolite content was determined by quantitative x-ray methods of the R+T sample fraction.

For -70+100M + -100M: Tremolite content was determined by quant x-ray method of the whole fraction.

a. assuming no tremolite in expanded vermi at all (0.2%)

b. assuming max. amount of tremolite in expanded Vm which can't be detected by x-rays accurately (0.5%)

CONSTRUCTION
PRODUCTS
DIVISION

PAGE 1

NUMBER: 50038
GROUP: Zonolite
DATE: 2/1/77
CHARGE NO.: S.O. 86-036 (71-[illegible])
REQUESTOR: F. W. Eaton
MARKETING or MANUFACTURING APPROVAL:
NAME: F. W. Eaton
APPROVED: [signature]

03627761

REQUEST FOR TECHNICAL SERVICE

PROBLEM TITLE: Moisture and bulk density determination - Libby #2 binder trials.

SIGNIFICANCE: Determine effect water addition (12.2 GPH) has on expanded vermiculite when water is used as a binder and applied at the product elevator and stoner discharge.

SPECIFIC OBJECTIVE:
Determine 1. % moisture
2. bulk density

SUGGESTED APPROACH:

DEADLINE (Last day information will be of value): As soon as possible. Part of total water binder evaluation and should be part of the personnel fiber count, drop test fiber count and tremolite determination.

DETAILS OF PROBLEM:

Determine moisture and bulk density on samples CL2, EL2 and SL2. Material is being shipped from Weedsport in 3 cu. ft. plastic bags.

ACCEPTED BY RESEARCH DEPT.: [signature] DATE: 2/2/77

ASSIGNED TO: S Vaughan

ADDITIONAL COPIES: Original to Library, H.C.Duecker, H.A.Eschenbach, F.W.Eaton, CPD-T&A, File: ~~86-036~~ [illegible]

CONFIDENTIAL

20152829

068FTX02085

REQUEST FOR TECHNICAL SERVICE

NUMBER:	50038
GROUP:	ZONOLITE
ACTUAL COST:	$35.00
REPORTING DATE:	February 11, 1977

03627762

SUMMARY:

Three samples of Libby #2 vermiculite were determined for moisture content. Bulk density evaluations showed no difference before and after the moisture removal. The moisture content of the stoner discharge material is considerably higher than the control. The product elevator material shows slightly more moisture than the control.

EXPERIMENTAL:

A representative portion of vermiculite was taken from each bag and quartered into 100 g samples. The samples were heated in the oven at 125°C. overnight, to determine the moisture content. The bulk density of the material as received, and after heating, was determined.

Sample	Moisture Content (%)	Bulk Density (PCF) as received	Bulk Density (PCF) after heating
CL2	0.9	5.5	5.4
EL2	1.2	5.7	5.6
SL2	3.1	6.0	5.9

Reference: 98162P

Steven Vaughan

SV:mlr

20152830

068FTX02086

CAMBRIDGE

03627763

TO: J. W. Wolter

DATE: January 6, 1977

FROM: Julie C. Yang

SUBJECT: Tremolite Content in Libby Vermiculite Composites

CC: E. S. Wood
R. L. Oliverio/Libby
H. C. Duecker
F. W. Eaton
File: 71-048

Recently we have determined the tremolite content in Libby #2 composite for the electrostatic spray studies, and found tremolite was in the range around 2.5% which showed a remarkable decrease over the #2 composite we had a year ago. The sample obtained in December 1975 showed about 5% tremolite (report on Libby Ore Evaluation 2/23/76).

If you would like to have the tremolite fiber content of composites of all sizes checked occasionally, we would be glad to do it. The cost of fiber determination for size 1 and 2 is about $80.00 each, and for size 3, 4 and 5 is around $120 per sample.

Julie C. Yang

JCY:mlr

20152831

PRODUCTS

DIVISION

CONFIDENTIAL

PAGE 1

GROUP: Zeolite

DATE: June 25, 1976

CHANGE NO.: 71-048

REQUESTOR: R. H. Locke

MANUFACTURING APPROVAL:

NAME: H. A. Brown

APPROVED: _____

03627764

REQUEST FOR TECHNICAL SERVICE

PROBLEM TITLE: Perform quantitative analysis of one-bag samples of MK-4 and MK-5 (already in Cambridge)

SIGNIFICANCE: To determine amount of tremolite present.

SPECIFIC OBJECTIVE: Approximation of analysis results which might be obtained by a laboratory facility other than ours.

SUGGESTED APPROACH: Perform analysis twice. The first approach utilize methods, equipment, and procedures which CPD laboratory personnel would use based on experience, etc. Second approach to be that (or those) which an outside facility would use (possibly same as CPD; if different from CPD, possibly more than a single alternate).

DEADLINE (Last day information will be of value): There is not now an identified deadline. However results are requested within a 30 to 45 day period if practical.

DETAILS OF PROBLEM: H. C. Duecker is familiar with all pertinent details.

ACCEPTED BY RESEARCH DEPT.: Julia C Yang DATE: 7/5/76

ASSIGNED TO: J. C. Yang / Steven Vaughan

20152832

ADDITIONAL COPIES: Original to Library, B.A.Blessington, H.C.Duecker, F.W.Eaton, P.E.Korenberg, R.H.Locke, R.A.Merther, L.S.Shu, B.R.Williams, J.W.Wolter, E.S.Wood, R.M.Vining, CPD-T&A, File: 71-048

CONFIDENTIAL

REQUEST FOR TECHNICAL SERVICE:

CONFIDENTIAL

NUMBER: 49189
GROUP: ZONOLITE
ACTUAL COST: $2500.00
REPORTING DATE: May 26, 1977

03627765

SUMMARY:

Three bags of standard MK-4 product from plant locations in California from Los Angeles, Santa Ana, and Newark), and four MK-5 samples (from Los Angeles, Santa Ana, and Omaha) have been examined for their tremolite content.

All seven samples as received showed no detectable tremolite fiber content by x-ray determinations (our detection limit for tremolite is 0.2%). However, the materials were fractioned; glass fibers were mostly retained on a +6 mesh screen, vermiculite was floated off; most of the plaster of Paris was dissolved in water; and, CELIF fibers and organic matter were burnt off. The concentrated fines, collected on Millipore filter of 0.45 μ, showed the presence of trace amounts of tremolite fiber in two of the trace MK-4 samples (Santa Ana and Newark). By petrographic microscopic examination, this was estimated to be less than 0.015% of the total sample.

The concentrates were then submitted to Arthur D. Little, Inc., for transmission and scanning electron microscopic analysis (TEM and SEM), selected area electron diffraction (SAED) and energy dispersive x-ray analysis (EDAX).

By these sophisticated and time-consuming instrumental analyses, the amphibole fibers were positively identified and analyzed. On a mass basis, it was found to be less than 0.006% of the concentrates which corresponded to 1.7 ppm * (Santa Ana) and 4.1 ppm (Newark) of the total MONOKOTE® sample weight.

EXPERIMENTAL:

Concentration

The concentration procedure of MONOKOTE is shown in Figure 1. The results are tabulated as follows:

---

* parts per million, or 0.00017%.

20152833

REQUEST FOR TECHNICAL SERVICE

NUMBER: 49189
GROUP: ZONOLITE
ACTUAL COST: $2500.00
REPORTING DATE: May 26, 1977

Fraction	Description	Material Present	% by weight in each Fraction						
			MK-4			MK-5			
			L.A. (8/76)	S.A. (8/76)	Newark (8/76)	Omaha new (8/76)	Omaha old	S.A. (10/76)	L.A. (10/76
1	Soluble	plaster of Paris	28.5	40.2	46.0	33.3	37.2	43.6	40.4
2	+6 Mesh	glass fiber	57.8	56.9	47.1	56.0	49.2	49.1	55.3
3	-6 +50	glass fiber, expanded Vm, some insoluble plaster							
4	Fines	some insoluble plaster, fine Vm. and tremolite (?), gypsum	13.7	2.9	6.9	10.7	13.6	7.3	4.3
			100.0%	100.0	100.0	100.0	100.0	100.0	100.0

X-Ray Diffraction Analysis

No detectable tremolite found in any of the fractions of the seven samples.

REQUEST FOR TECHNICAL SERVICE:

CONFIDENTIAL

NUMBER:	49189
GROUP:	ZONOLITE
ACTUAL COST:	$2500.00
REPORTING DATE:	May 26, 1977

Petrographic Microscopic Examination

03627767

Based on the characteristic refractive indices and optical properties of vermiculite and tremolite fibers, using the liquid immersion technique, a trace of tremolite was found in the -50M +0.45 μ portion of Santa Ana MK-4, and Newark MK-4 samples.

Analysis by Arthur D. Little, Inc.

Even though the original request made by R. H. Locke was on one MK-4 and one MK-5 sample, we have decided to do several more since the product from each plant looked and behaved very differently. The MK-4 from Newark was very dense and the vermiculite present was poorly expanded in comparison with the others. Product from Santa Ana was very bulky and the plaster of Paris present in the composition dissolved more readily than the others.

The two concentrated samples suspected to have tremolite fibers were submitted to Arthur D. Little for fiber characterization and counting on transmission micrographs (Figures 2 and 3). Each fiber being counted was analyzed by SAED (selected area electron diffraction) to determine the structure of the fiber. It was found that 25-40% of fibers did not yield an SAED pattern indicating the fiber was amorphous, mostly organic and glass fibers. The breakdown of the fiber types and amounts is listed in Table 1.

Scanning electron micrographs were also taken on some of the fibers. They are shown in Figures 4 and 5, and energy dispersive x-ray analysis (EDAX) was employed to analyze the elements present in each fiber. The results are shown in Table 2.

CONCLUSIONS and COMMENTS:

The conclusion reached by A. D. Little, Inc. was that the amphibole fiber content, on a mass basis, corresponded to less than 0.006% of the supplied concentrated sample. Letter from Dr. E. Peters of ADL is attached. Computing the amphibole content in the MONOKOTE samples from Santa Ana and Newark, this corresponds to less than 1.7 ppm and 4.1 ppm, respectively. The level of tremolite fiber present was extremely low.

Julie C. Yang

JCY:mlr

attachment

20152835

068ETX02091

REQUEST FOR TECHNICAL SERVICE: **CONFIDENTIAL**

NUMBER: 49189
GROUP: ZONOLITE
ACTUAL COST: $2500.00
REPORTING DATE: May 26, 1977

TABLE 1 - Fiber analysis by TEM (A.D.Little)

03627768

Fiber Observed	Sample 22281-1 Fines Fraction from Santa Ana, MK-4 Sample	Sample 22281-2 Fines Fraction from Newark, MK-4 Sample
Total fibers observed	104	54
% Amphibole	6	4
% Other Mineral (mostly gypsum)	33.5	35
% Ambiguous Mineral (with insufficient data for positive identification)	34.5	22
% Amorphous (organic, glass fiber)	26	39
	100%	100%

TABLE 2 - EDAX Microchemical Analysis of Fibers Observed by Scanning Electron Microscopy (A.D.Little)

		Relative X-ray Intensity			
Sample 22281-1		Strong	Medium	Weak	Probable I.D.
Fiber 1	Figure 6a	Al	S	Mg	
Fiber 2	Figure 6b	Si,Al	Mg,Ca,S	Fe,K	amphibole or glass
Fiber 3	Figure 4a	Al	-	Ca,S,Si	gypsum (?)
Fiber 4	Figure 4b	Si,Al,Mg,S	Ca,Fe	K	amphibole or glass
Sample 22281-2					
Fiber 5	Figure 3	S,Ca,Al			gypsum

20152836

Arthur D. Little, Inc. ACORN PARK · CAMBRIDGE MASSACHUSETTS 02140 · (617) 864-5770

April 5, 1977

03627769

Dr. Julie C. Yang
Manager, Research Technologies
Construction Products Division
W. R. Grace & Co.
62 Whittemore Avenue
Cambridge, Mass. 02140

Dear Julie: C76494

As we discussed during your visit on March 11, 1977, low magnification transmission electron microscope photographs have been obtained from two representative grid pore openings of samples 22281-1 and 22281-2 to permit an estimate of the percentage of mass attributable to fibers, in particular, amphibole fibers. A previous analysis of these samples, reported on January 24, 1977, identified the presence of fibers, most of which were mineral. These results can be summarized as follows:

	Santa Ana 22281-1	Newark 22281-2
Fibers observed	104	54
Percent amphibole	6	4
Percent other mineral (mostly gypsum)	34	35
Percent ambiguous mineral	35	22
Percent amorphous (organic, glass fiber)	26	39

As some of the ambiguous mineral category may be amphibole, it is prudent to estimate a maximum amphibole fiber content of 10 percent. Due to a slightly larger fiber size, the amphibole fiber volume is about 15 percent of the total fiber volume, which corresponds to 1.6×10^{-12} cm^3 per grid pore opening.

To estimate the relative amount of fibrous material present in the samples, low magnification TEM photographs were obtained from two representative pore openings of both samples. These were assembled into

20152837

C68ETX02093

03627770

Dr. Julie C. Yang
W. R. Grace & Co.

montages, which covered entire pore openings. Particle volumes per pore opening were calculated for the two montages prepared for sample 22281-1A (exhibiting the heaviest particle loading) from the projected surface area and an estimated thickness of each particle, as follows:

0.2μm - particles showing electron beam penetration over whole area

0.5μm - particles showing electron beam penetration at edges

1-2μm - electron opaque particles

From these estimates, the ratio of fiber volume to total particle volume was estimated to be 0.04 percent (0.006 percent for amphibole fibers). For the assumption that the densities of all particles are equivalent, these percentages apply on a mass basis, as well.

From this analysis, we conclude that the amphibole fiber content, on a mass basis, corresponds to less than 0.006 percent of the supplied sample, which represented the insoluble residue fraction of a leached Monokote sample. This estimate should be reliable within a factor of two times.

Please contact me if you have any questions.

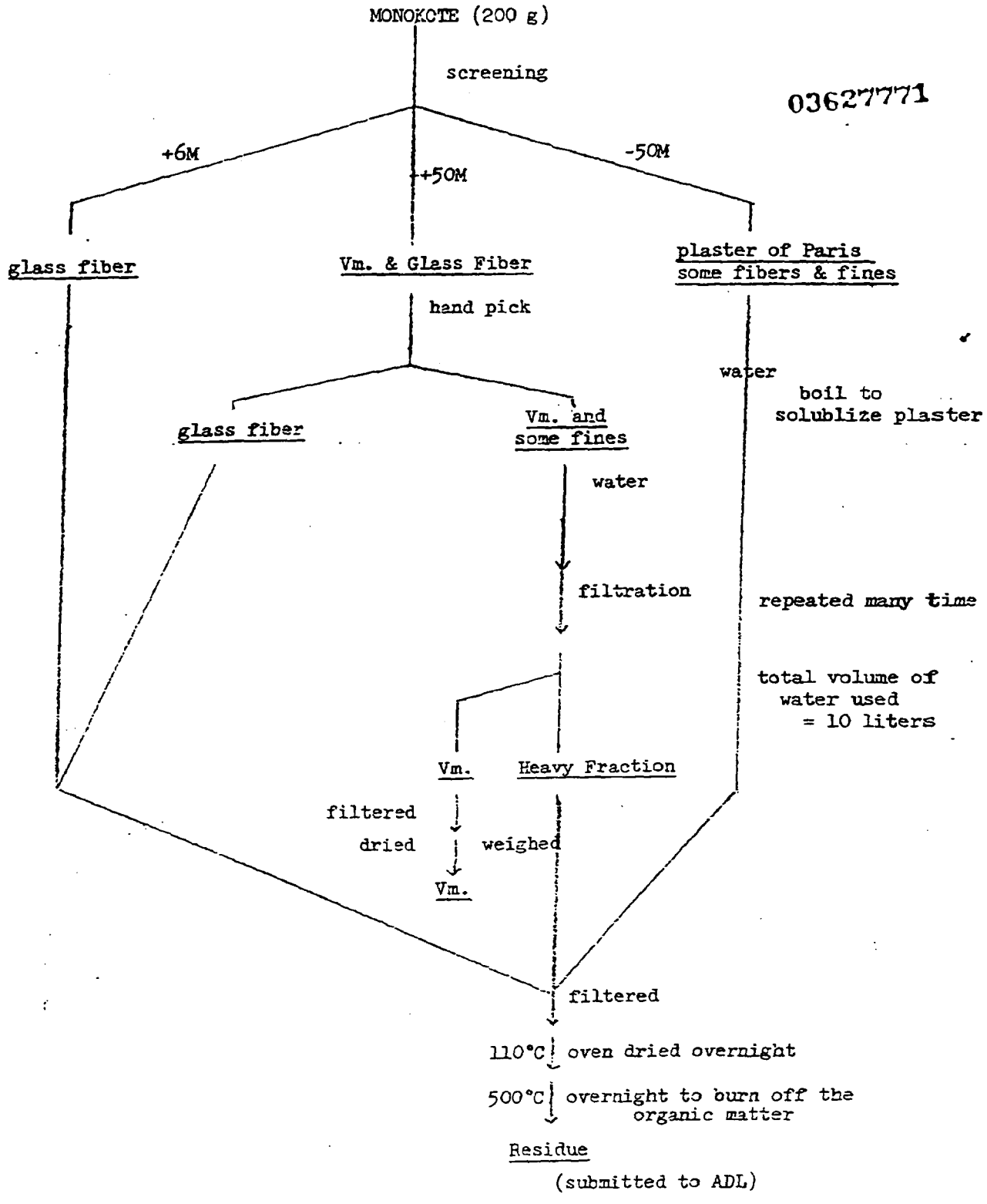
Very truly yours,

Edward T. Peters

/rdl

20152838

FIGURE 1 - CONCENTRATION OF FINES IN MONOKOTE®



J.C.Yang:mlr
5/26/77

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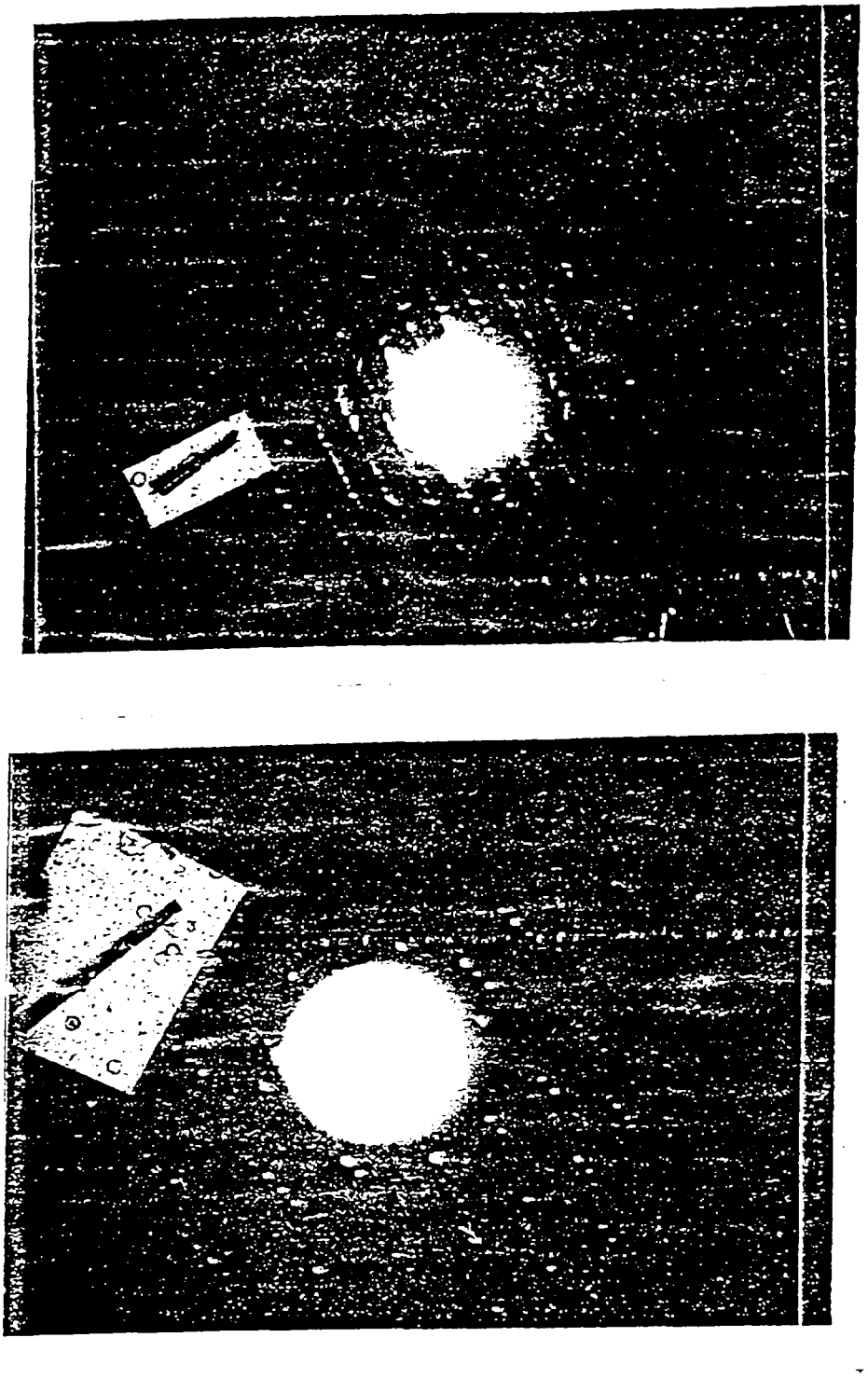

Figure 2. Transmission Electron Image of Fibrous Particles and Corresponding SAED Patterns, Sample 22281-1; 10,000x.

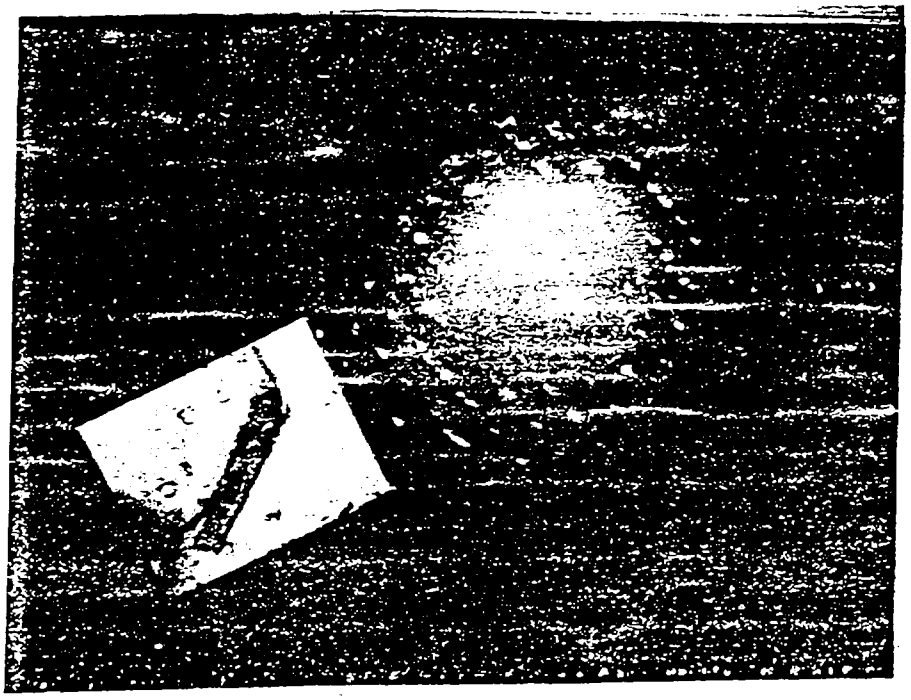

Figure 3. Transmission Electron Image of Fibrous Particles and Corresponding SAED Pattern, Sample 22281-2; 10,000x.

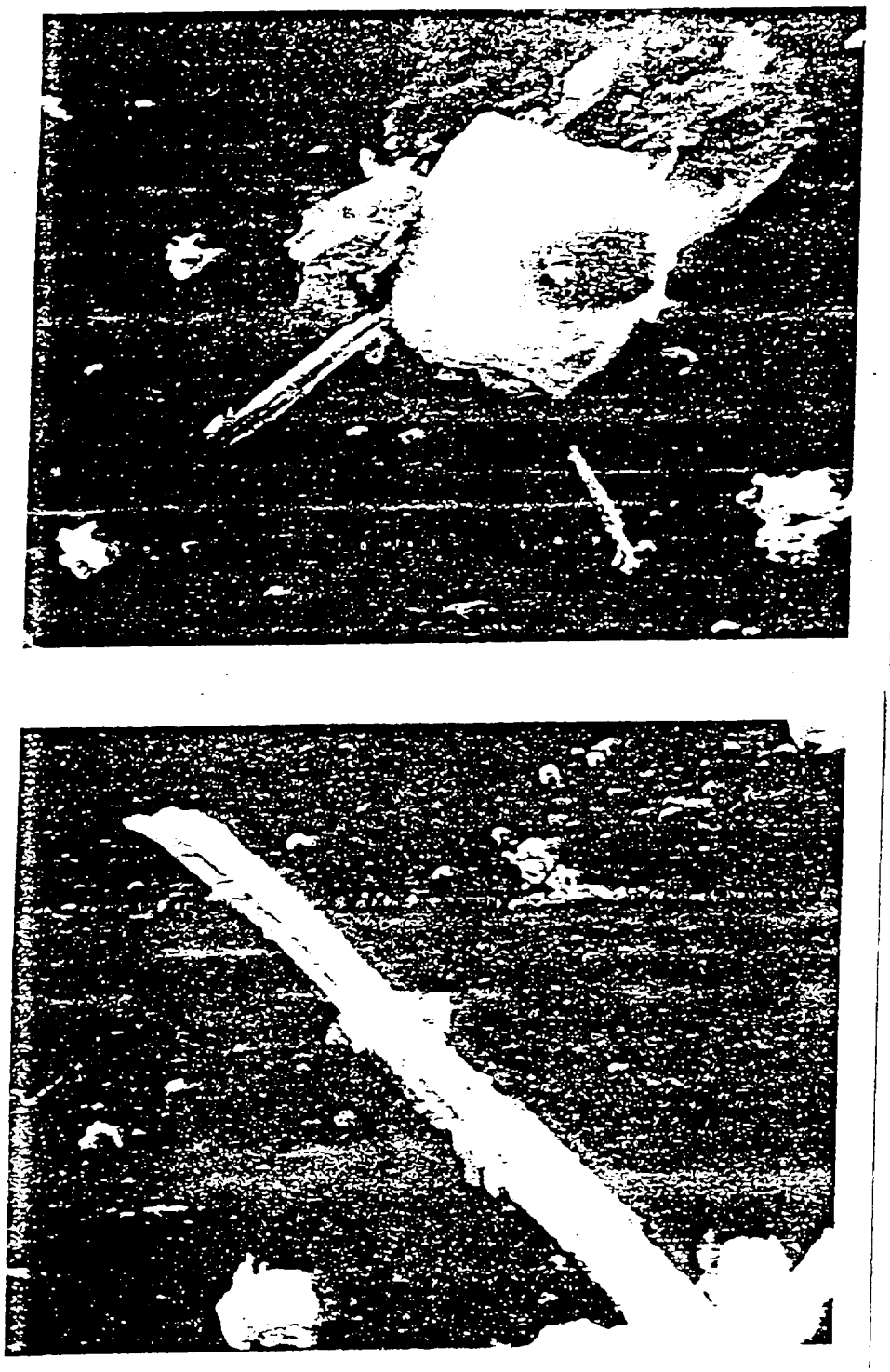

Figure 4. Scanning Electron Micrographs of Fibrous Particles in Sample 22281-1
a) 5500x, b) 5500x

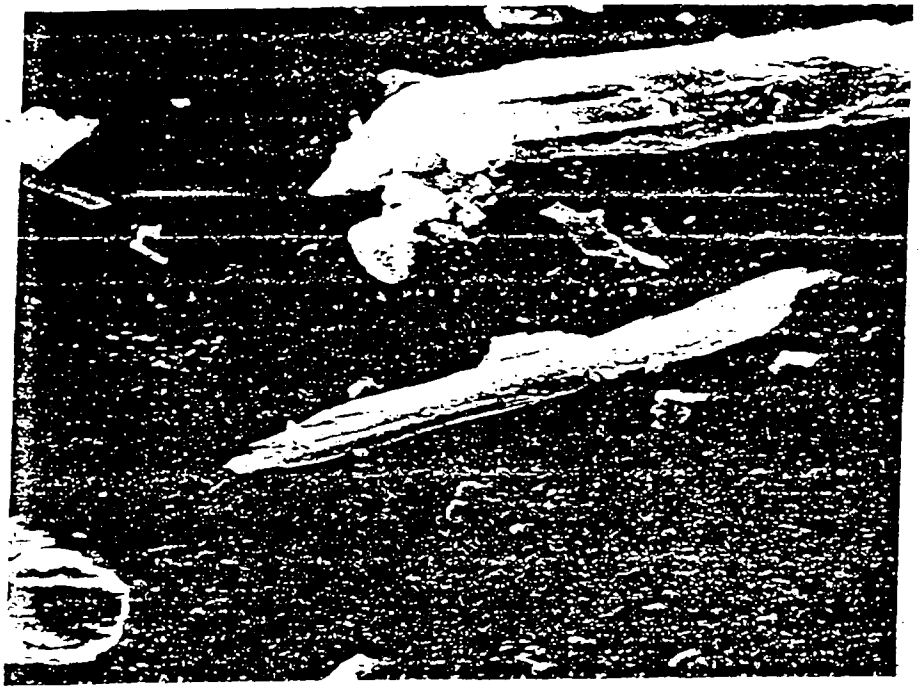

Figure 5 | Scanning Electron Micrograph of a Fibrous Particle in Sample 22281-2, 5500x.

Figure 6) Scanning Electron Micrographs of Fibrous Particles in Sample 22281-1. a) 2400x, b) 1100x

GRACE

Construction Products Division

03627777

CHARACTERIZATION AND PREPARATION OF RESPIRABLE SIZED TREMOLITE FIBER AND VERMICULITE FOR ANIMAL STUDIES

by: Julie C. Yang

April 8, 1976

20152845

CAMBRIDGE

03627778

TO: H. C. Duecker

DATE: April 8, 1976

FROM: Julie C. Yang

SUBJECT: Characterization and Preparation of Respirable Sized Tremolite Fiber and Vermiculite for Animal Studies

CC: H. A. Brown
J. W. Wolter
H. A. Eschenbach
R. H. Locke
File: 71-048

PURPOSE

The objectives of this study are to find out the size distribution and concentration of the respirable size fibers and vermiculite on the air filter collected by the the Industrial Hygiene and Environmental Health group in the field, and to prepare the samples corresponding as closely as possible to these air filter material, for animal studies.

AIR FILTER STUDY

Several randomly collected air samples from Libby at fairly long time intervals were collected for fiber contents and submitted to Arthur D. Little for sizing and distribution studies.

Two samples were sent:

Sample No.	Collecting Time	Fiber Count (Optical/40 Fields)
22260P-1	248 mins.	0.18 Fiber/cc air
22260P-2	300 mins.	2.15 Fiber/cc air

The results from Arthur D. Little are shown in Tables 1 and 2, Figures 1 - 3; and conclusions reached are summarized as follows:

1) On the air filter the respirable sized vermiculites and tremolite fibers are roughly in 50-50% ratio.
2) The respirable size tremolite fibers are mostly less than 10 microns ($< 8\% > 10\,\mu$ size), and the geometric mean length of the fibers is around 3.1 μ.
3) The respirable size vermiculites are also less than 10 μ, having an average size about 5 μ.
4) The aspect ratio of the fibers is in the range of 11 to 15 μ.
5) Computation shows that the fiber counting with SEM (scanning electron microscope)@ 20,000 magnification. The total numbers of fibers found per unit area (1 cm^2) is about seven times in number of the fibers found by optical microscope counting at 400 magnification.

20152846

To: H. C. Duecker
From: J. C. Yang

Re: Animal Studies
April 9, 1976

03627779

SAMPLE PREPARATIONS

After we characterized what we have on the air filter, attempts were made to prepare both respirable sized vermiculite and tremolite fibers as closely as possible to those found on the air filter.

From previous research work (report on Libby Ore Evaluation - Ore Impurities, 2/23/76) we have found that Libby #2 vermiculite product has the highest tremolite fiber content in the order of 5% by weight. Since the sizes of #2 are fairly and easily to be handpicked, it is used as a starting source for both tremolite and vermiculite.

The tremolite fiber bundles picked out from Libby #2 are fairly clean and free of rocks, greyish in color, soft, and sometimes waxy in touch. They broke down easily to fine fibrils when degraded, which looked extremely similar to those found on the filter or floating in air in the Libby operation, which are quite different than the tremolite found in associated veins in rock form; they are generally harder and harsher, most of which were removed in the floatation process.

1) Tremolite Fiber

a) Cleaning

Tremolite fiber bundles were hand-picked from Libby #2 product, cleaned with acetone and then distilled water. The bundles were then opened with Waring Blender for 2 minutes at high speed, filtered and dried in the oven at 105°C. for about four hours.

b) Milling

The oven-dried material was Spec-milled in 0.5 g batch for a total of 45 seconds; but after each 10 seconds milling interval the mill was stopped and the material reruffled to avoid excessive packing.

The Spec-milled samples were then chilled in dry ice-acetone batch, chilling at low temperature increases the brittleness of the fibers and makes them easier to be pulverized. The chilled fibers were subjected to a Wiley mill with a built-in 60 mesh screen, a mill which has been designed especially for milling fibers. The Wiley milling was repeated another three times. Between runs the material has to be chilled again thoroughly with dry ice.

c) Sedimentation

0.8 g of the Wiley milled sample (mostly 2-4 μ in size, some up to 30 μ with some bundles under light microscope) was dispersed in two liters of distilled water, allowed to stand for 20 minutes; then, decant the cloudy solution into 250 ml or 500 ml graduated cylinders which were employed as sedimentation columns, and dilute the solution to twice its volume with distilled water. The solutions in each column were lightly stirred and allowed to settle for twenty minutes. The cloudy solution was then filtered by an HA type Millipore filter of 0.45 μ. However, the filterate looked extremely clear and showed some small particles under the microscope.

20152847

To: H. C. Duecker
From: J. C. Yang

Re: Animal Studies
April 9, 1976

The solid collected from the beaker and the column were recombined and treated with another 2 liters of distilled water, poured into columns and allowed to stand overnight. The cloudy solution was again decanted and filtered through the Millipore. Coarse solid remained at the bottom of the column from the second sedimentation, was filtered and saved for future remilling. The five fibers collected on the top of the Millipore were then examined by light microscope. It was found most of the particles were around 2 μ, and a few long fibers up to 20 μ.

d) Cleaning and Resizing

The finished crude product from step c. was redispersed in the order of 2 g/4 liter distilled water, and allowed to stand in columns for over half an hour. The decanted cloudy solution (about twice as dense as solution in step c.) was then filtered through Millipore filter. The solid left at the bottom of the column was dispersed again, ultrasonically, for 2 minutes in 400 ml water. The milky solution was then diluted to another 4 liters and allowed to settle in columns for a final 20 minutes. The fines were collected on Millipore by filtering the decanted liquid, dried as examined by light microscope. The product has mostly 2 μ in size, very few larger fibers but a few up to 10 μ. The solid remained from decantation was again filtered and saved for future remilling.

2) Vermiculite

a. Cleaning

The vermiculite platlets were also hand-picked from Libby #2 product, cleaned in Soxhlet extractor with isopropyl alcohol, then acetone, and finally water to remove all the trace of organic contaminants used in the flotation process; then oven-dried at 105°C. for several hours.

b. Milling

The oven-dried vermiculite was then chilled with acetone and dry-ice mixture, Spec-milled in 2 g batches for 10 minutes. At the end of 5 minutes, the mill was stopped and the material was reruffled.

c. Screening

The milled sample was screened with 325 mesh screen. The -325 mesh product showed the desirable respirable size. Most of the particles were 2 - 4 μ. Some large plates were about 10 - 15 μ. The +325 mesh material was also collected and saved for future remilling.

To: H. C. Duecker
From: J. C. Yang

Re: Animal Studies
April 9, 1976

03627781

3) Proportioning

5 g of tremolite and 5 g of vermiculite, prepared from step 1) and 2) respectively, were carefully weighed out on a semimicro balance, and then transferred to a 4 oz. size wide-mouth glass bottle in which some silver wires were added to break up the powder surface when mixed on a roller mill. The mixing was carried out for about 16 hours. Because of the morphology and density difference, it will be suggested to Dr. Smith that when this sample is being used for animal study, an appreciable quantity (such as 1 or 2 grams) is taken, then dispersed in the saline medium ultrasonically, prior to use. The purpose of doing this will eliminate the localized inhomogenity and selectiveness of a very small sample.

4) Characterization

The respirable-sized fibers (2260P-4 and 22250P-5) have been sent to A. D. Little for sizing and comparison with the fiber found on the air filter. The results are also shown in Tables 1 and 2, Figures 7 and 8. Scanning electron micrographs of these materials are shown in Figures 9 - 10.

Results from A. D. Little and our own microscopic sizing indicated that the respirable size fibers and vermiculite which we prepared are very similar to those on the air filter. However, sample 22260P-4 is a fiber sample of finer size, extremely time-consuming to obtain in large quantities. We have then taken a different approach to obtain 22260P-5 which is slightly coarser than 22260P-4. The two samples of 8 grams each we have submitted to Dr. W. Smith are:

1. 22260P-5 - respirable sized tremolite fiber
2. 22263P-2 - a mixture in 50-50% of respirable sized tremolite fiber (22260P-5) and vermiculite (22263P-1)

The final characterization of samples will be made by Walter McCrone Associates:

1. 22260P-5 respirable sized tremolite fiber
2. 22263P-1 respirable sized vermiculite
3. 22263P-3 a saline suspension of 22263P-2 will be prepared by W. Smith's group for animal studies.

20152849

To: H. C. Duecker
From: J. C. Yang

Re: Animal Studies
April 9, 1976

03627782

5) Sample Preparation for Animal Injection Studies

Dr. Smith's group has been preparing samples by dispersing 2 g of the solid in 40 ml 0.9 g saline solution in a 100 ml Erlenmeyer flask, then autoclaved for 15 minutes at 15 - 20 psi to sterilize the material. After it was cooled off, the mixture was shaken by hand and drawn into a syringe in 1 ml aliquot for injection.

By observing the preparations made with R. T. Vanderbilt sample (talc and tremolite mixture), solid settled very quickly in the saline solution immediately after shaking. Employing such technique, I would expect the animals got different doses of material depending on the technique of the operator and the rate of settling at that specific time. In addition, the fibers present may be in bundles or small balls not fully opened.

As a result, I have recommended the use of ultrasonic dispersion. The saline suspension after autoclaved should be subjected to a 10 minute sonic dispersion. It has been demonstrated the respirable sized material was suspended quite uniformly for an hour or more without settling. In case of any fiber balls or bundles present, they will be fully opened and dispersed, too.

Each animal will get 1 ml of the suspension which has 25 mg of the solid theoretically.

Julie C. Yang

JCY:mlr
attachments

20152850

TARGET SHEET

EPA REGION VIII

SUPERFUND DOCUMENT MANAGEMENT SYSTEM

DOCUMENT NUMBER: 495699

SITE NAME: LIBBY ASBESTOS

DOCUMENT DATE: 02/23/1976

DOCUMENT NOT SCANNED

Due to one of the following reasons:

- ❑ PHOTOGRAPHS
- ❑ 3-DIMENSIONAL
- ❑ OVERSIZED
- ❑ AUDIO/VISUAL
- ❑ PERMANENTLY BOUND DOCUMENTS
- ❑ POOR LEGIBILITY
- ❑ OTHER
- ❑ NOT AVAILABLE
- ✓ TYPES OF DOCUMENTS NOT TO BE SCANNED
  (Data Packages, Data Validation, Sampling Data, CBI, Chain of Custody)

DOCUMENT DESCRIPTION:

TABLES 1 & 2, FIGURES 1 - 10

Contact the Superfund Records Center to view available document.
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495726

ADMINISTRATIVE RECORD

CONSTRUCTION PRODUCTS DIVISION

REC'D APR 5 1978 C.P.D. ENG.

PAGE 1

REQUEST FOR TECHNICAL SERVICE

03643236

NUMBER: #69515S(Addendum)
GROUP: BPD
DATE: 3/14/78
CHARGE NO.: 71-174
REQUESTOR: R. C. Ericson
MARKETING or MANUFACTURING APPROVAL:
NAME: R. C. Ericson
APPROVED: R C Ericson

PROBLEM TITLE: Determine % Tremolite in samples of Libby #1 "Attic" taken from the volume yield test run on a Model A furnace in Chicago 3/8/78.

SIGNIFICANCE: Information needed as part of our continuing study of how to reduce tremolite contamination in the finished product.

SPECIFIC OBJECTIVE: 180 cu.ft. of product shipped to Weedsport. Fred Eaton will run simulated Attic Fill trials — E. S. Wood memo 3/2/78 attached.

SUGGESTED APPROACH:

DEADLINE (Last day information will be of value):

DETAILS OF PROBLEM:

Samples Furnished

(1) Test #2 - Samples 1, 2 & 3 to be composited together - Finished product

(2) One Sample, screen unders

Data Attached

(1) Data Summary Sheets (3)

(2) Analysis of screened unders - Cambridge Q. C.

ACCEPTED BY RESEARCH DEPT.: J. C. Yanno DATE: 3/21/78

ASSIGNED TO: S. Vaughan / J. P. Hackler

ADDITIONAL COPIES: Original to Library H. C. Duecker, E. S. Wood, F. W. Eaton, R. E. Schneider, J. W. Wolter and R. C. Ericson

CONFIDENTIAL

15035669

REQUEST FOR TECHNICAL SERVICE

NUMBER: 69515 Supplementary
GROUP: BPD
ACTUAL COST: $230.00
REPORTING DATE: April 4, 1978

03645237

SUMMARY:

Two samples were received for tremolite analysis.

DATA AND ANALYSIS:

The results are:

I.D. No.	Description	% unfloatable	% Tremolite
1)	Composite of sample from Test #2 - Finished product	1.5	.02
2)	Screen unders	100.0	4.6

Julie C. Yang

JCY:mlr

15035670

CAMBRIDGE

TO: H. C. Duecker

DATE: February 23, 1976

FROM: Julie C. Yang

SUBJECT: Libby Ore Evaluation - Ore Impurities

CC: H. A. Brown
J. W. Wolter
R. L. Oliverio/Libby
R. J. Kujawa/Libby
G. G. Vaplon/Libby
O. F. Stewart/Enoree
R. H. Locke
J. L. Young
File: 71-048

PURPOSE

The objective of this investigation is to determine the tremolite content for each of the three mill circuits and end products at Libby.

SAMPLE SELECTION

Samples have been collected by G. Vaplon:

(a) material which entered the circuit,
(b) material which came out of the circuit,
(c) screened plant products as control and comparison with (a) & (b).

Fourteen materials were received:

(1)	Clean Conc.	8 x 20		
(2)	Rough Conc.	8 x 20		
(3)	Rough Conc.	20 x 65		
(4)	Clean Conc.	20 x 65		
(7)	Rough Feed	8 x 20		
(8)	Clean Feed	8 x 20		
(9)	Rough Feed	20 x 65		
(10)	Clean Feed	20 x 65		
(11)	#1 Composite			
(12)	#2 Composite			
(5)	#3 Composite			
(6)	#4 Composite			
(13)	#5 Composite			
(14)	Humphrey Sizer Concrete		12/3/75	9:00 a.m.

EXPERIMENTAL

I) Humphrey Sizer

1. Separation

The rock and fiber were separated from the vermiculite plates by hand-picking.

2. Method of Analysis

Each portion has been weighed carefully and then x-rayed for their mineral content.

To: H. C. Duecker
From: Julie C. Yang
Feb. 23, 1976

Libby Ore Evaluation - Ore Impurities

03627S01

CONCLUSIONS

1. The possible tremolite content of end products of each size and of concentrates from the three circuits are:

Circuit	Tremolite Contents, percent Range	Mean
Humphrey Sizer	2.70 - 2.72	2.71
8 x 20		
Rough concentrate	0.21 - 0.71	0.46
Clean concentrate	0.10 - 0.59	0.35
20 x 65		
Rough concentrate	0.4 - 0.86	0.63
Clean concentrate	0.74 - 1.20	0.97
End Product		
Composites #1	1.67 - 2.17	1.92
#2	4.72 - 5.22	4.97
#3	0.41 - 0.89	0.65
#4	0.52 - 1.00	0.76
#5	3.45 - 3.97	3.71

2. Based on the experimental data, the approximate amount of tremolite present in tons per day, out of each of the three circuits, will be as follows:

Circuit	Total Materials out of the circuit (tons/day)*	Mean Tremolite Content (tons/day)
Humphrey Sizer	220	5.96
8 x 20	295	1.16
20 x 65	513	4.10

*based on 22 hours in a day.

3. The #2 composite showed the highest tremolite content (even more so than #5), and the fibers present are mostly in heavy bundle form, visible to the eye. This fact is also true for the material in the 8 x 20 circuit and other coarse end products #1 and #3. The tendency of fiber balling in the 20 x 65 circuit shows that the fibers are more opened or in thinner bundles in addition to some extra fines distributed throughout the end products #4 and #5, which will lead to the belief that there is some degree of down screening.

20152824

Attachment #58

GRACE

Construction Products Division

CHARACTERIZATION AND PREPARATION OF RESPIRABLE SIZED TREMOLITE FIBER AND VERMICULITE FOR ANIMAL STUDIES

by: Julie C. Yang

April 8, 1976

CAMBRIDGE

03627778

TO: H. C. Duecker

DATE: April 8, 1976

FROM: Julie C. Yang

SUBJECT: Characterization and Preparation of Respirable Sized Tremolite Fiber and Vermiculite for Animal Studies

CC: H. A. Brown
J. W. Wolter
H. A. Eschenbach
R. H. Locke
File: 71-048

PURPOSE

The objectives of this study are to find out the size distribution and concentration of the respirable size fibers and vermiculite on the air filter collected by the the Industrial Hygiene and Environmental Health group in the field, and to prepare the samples corresponding as closely as possible to these air filter material, for animal studies.

AIR FILTER STUDY

Several randomly collected air samples from Libby at fairly long time intervals were collected for fiber contents and submitted to Arthur D. Little for sizing and distribution studies.

Two samples were sent:

Sample No.	Collecting Time	Fiber Count (Optical/40 Fields)
22260P-1	248 mins.	0.18 Fiber/cc air
22260P-2	300 mins.	2.15 Fiber/cc air

The results from Arthur D. Little are shown in Tables 1 and 2, Figures 1 - 3; and conclusions reached are summarized as follows:

1) On the air filter the respirable sized vermiculites and tremolite fibers are roughly in 50-50% ratio.

2) The respirable size tremolite fibers are mostly less than 10 microns ($< 8\% > 10\,\mu$ size), and the geometric mean length of the fibers is around 3.1 μ.

3) The respirable size vermiculites are also less than 10 μ, having an average size about 5 μ.

4) The aspect ratio of the fibers is in the range of 11 to 15 μ.

5) Computation shows that the fiber counting with SEM (scanning electron microscope)@ 20,000 magnification. The total numbers of fibers found per unit area (1 cm^2) is about seven times in number of the fibers found by optical microscope counting at 400 magnification.

20152846

068ETX02102

To: H. C. Duecker
From: J. C. Yang

Re: Animal Studies
April 9, 1976

03627779

SAMPLE PREPARATIONS

After we characterized what we have on the air filter, attempts were made to prepare both respirable sized vermiculite and tremolite fibers as closely as possible to those found on the air filter.

From previous research work (report on Libby Ore Evaluation - Ore Impurities, 2/23/76) we have found that Libby #2 vermiculite product has the highest tremolite fiber content in the order of 5% by weight. Since the sizes of #2 are fairly and easily to be handpicked, it is used as a starting source for both tremolite and vermiculite.

The tremolite fiber bundles picked out from Libby #2 are fairly clean and free of rocks, greyish in color, soft, and sometimes waxy in touch. They broke down easily to fine fibrils when degraded, which looked extremely similar to those found on the filter or floating in air in the Libby operation, which are quite different than the tremolite found in associated veins in rock form; they are generally harder and harsher, most of which were removed in the floatation process.

1) Tremolite Fiber

a) Cleaning

Tremolite fiber bundles were hand-picked from Libby #2 product, cleaned with acetone and then distilled water. The bundles were then opened with Waring Blender for 2 minutes at high speed, filtered and dried in the oven at 105°C. for about four hours.

b) Milling

The oven-dried material was Spec-milled in 0.5 g batch for a total of 45 seconds; but after each 10 seconds milling interval the mill was stopped and the material reruffled to avoid excessive packing.

The Spec-milled samples were then chilled in dry ice-acetone batch, chilling at low temperature increases the brittleness of the fibers and makes them easier to be pulverized. The chilled fibers were subjected to a Wiley mill with a built-in 60 mesh screen, a mill which has been designed especially for milling fibers. The Wiley milling was repeated another three times. Between runs the material has to be chilled again thoroughly with dry ice.

c) Sedimentation

0.8 g of the Wiley milled sample (mostly 2-4 μ in size, some up to 30 μ with some bundles under light microscope) was dispersed in two liters of distilled water, allowed to stand for 20 minutes; then, decant the cloudy solution into 250 ml or 500 ml graduated cylinders which were employed as sedimentation columns, and dilute the solution to twice its volume with distilled water. The solutions in each column were lightly stirred and allowed to settle for twenty minutes. The cloudy solution was then filtered by an HA type Millipore filter of 0.45 μ. However, the filterate looked extremely clear and showed some small particles under the microscope.

20152847

To: H. C. Duecker
From: J. C. Yang

Re: Animal Studies
April 9, 1976

The solid collected from the beaker and the column were recombined and treated with another 2 liters of distilled water, poured into columns and allowed to stand overnight. The cloudy solution was again decanted and filtered through the Millipore. Coarse solid remained at the bottom of the column from the second sedimentation, was filtered and saved for future remilling. The five fibers collected on the top of the Millipore were then examined by light microscope. It was found most of the particles were around 2 μ, and a few long fibers up to 20 μ.

d) Cleaning and Resizing

The finished crude product from step c. was redispersed in the order of 2 g/4 liter distilled water, and allowed to stand in columns for over half an hour. The decanted cloudy solution (about twice as dense as solution in step c.) was then filtered through Millipore filter. The solid left at the bottom of the column was dispersed again, ultrasonically, for 2 minutes in 400 ml water. The milky solution was then diluted to another 4 liters and allowed to settle in columns for a final 20 minutes. The fines were collected on Millipore by filtering the decanted liquid, dried as examined by light microscope. The product has mostly 2 μ in size, very few larger fibers but a few up to 10 μ. The solid remained from decantation was again filtered and saved for future remilling.

2) Vermiculite

a. Cleaning

The vermiculite platlets were also hand-picked from Libby #2 product, cleaned in Soxhlet extractor with isopropyl alcohol, then acetone, and finally water to remove all the trace of organic contaminants used in the flotation process; then oven-dried at 105°C. for several hours.

b. Milling

The oven-dried vermiculite was then chilled with acetone and dry-ice mixture, Spec-milled in 2 g batches for 10 minutes. At the end of 5 minutes, the mill was stopped and the material was reruffled.

c. Screening

The milled sample was screened with 325 mesh screen. The -325 mesh product showed the desirable respirable size. Most of the particles were 2 - 4 μ. Some large plates were about 10 - 15 μ. The +325 mesh material was also collected and saved for future remilling.

To: H. C. Duecker
From: J. C. Yang

Re: Animal Studies
April 9, 1976

03627781

3) Proportioning

5 g of tremolite and 5 g of vermiculite, prepared from step 1) and 2) respectively, were carefully weighed out on a semimicro balance, and then transferred to a 4 oz. size wide-mouth glass bottle in which some silver wires were added to break up the powder surface when mixed on a roller mill. The mixing was carried out for about 16 hours. Because of the morphology and density difference, it will be suggested to Dr. Smith that when this sample is being used for animal study, an appreciable quantity (such as 1 or 2 grams) is taken, then dispersed in the saline medium ultrasonically, prior to use. The purpose of doing this will eliminate the localized inhomogenity and selectiveness of a very small sample.

4) Characterization

The respirable-sized fibers (2260P-4 and 22250P-5) have been sent to A. D. Little for sizing and comparison with the fiber found on the air filter. The results are also shown in Tables 1 and 2, Figures 7 and 8. Scanning electron micrographs of these materials are shown in Figures 9 - 10.

Results from A. D. Little and our own microscopic sizing indicated that the respirable size fibers and vermiculite which we prepared are very similar to those on the air filter. However, sample 22260P-4 is a fiber sample of finer size, extremely time-consuming to obtain in large quantities. We have then taken a different approach to obtain 22260P-5 which is slightly coarser than 22260P-4. The two samples of 8 grams each we have submitted to Dr. W. Smith are:

1. 22260P-5 - respirable sized tremolite fiber
2. 22263P-2 - a mixture in 50-50% of respirable sized tremolite fiber (22260P-5) and vermiculite (22263P-1)

The final characterization of samples will be made by Walter McCrone Associates:

1. 22260P-5 respirable sized tremolite fiber
2. 22263P-1 respirable sized vermiculite
3. 22263P-3 a saline suspension of 22263P-2 will be prepared by W. Smith's group for animal studies.

20152849

To: H. C. Duecker
From: J. C. Yang

Re: Animal Studies
April 9, 1976

03627782

5) Sample Preparation for Animal Injection Studies

Dr. Smith's group has been preparing samples by dispersing 2 g of the solid in 40 ml 0.9 g saline solution in a 100 ml Erlenmeyer flask, then autoclaved for 15 minutes at 15 - 20 psi to sterilize the material. After it was cooled off, the mixture was shaken by hand and drawn into a syringe in 1 ml aliquot for injection.

By observing the preparations made with R. T. Vanderbilt sample (talc and tremolite mixture), solid settled very quickly in the saline solution immediately after shaking. Employing such technique, I would expect the animals got different doses of material depending on the technique of the operator and the rate of settling at that specific time. In addition, the fibers present may be in bundles or small balls not fully opened.

As a result, I have recommended the use of ultrasonic dispersion. The saline suspension after autoclaved should be subjected to a 10 minute sonic dispersion. It has been demonstrated the respirable sized material was suspended quite uniformly for an hour or more without settling. In case of any fiber balls or bundles present, they will be fully opened and dispersed, too.

Each animal will get 1 ml of the suspension which has 25 mg of the solid theoretically.

Julie C. Yang

JCY:mlr

attachments

20152850

TABLE 1

SUMMARY OF LENGTH DATA

03627783

Range (μ)	No. 1 (Total No.) N	No. 1 Cum %	No. 2 N	No. 2 Cum %	22260-P4 N	22260-P4 Cum %	22260-P5 N	22260-P5 Cum %
<0.3	2	4	0	0	0	0	0	0
0.3-0.4	6	14	1	1	1	1	0	0
0.4-0.5	4	21	1	2	4	4	1	1
0.5-0.6	6	32	1	2	3	7	2	3
0.6-0.7	0	32	2	4	5	12	0	3
0.7-0.8	7	44	5	8	3	14	3	5
0.8-0.9	2	47	4	11	3	17	3	9
0.9-1.0	1	49	0	11	4	20	2	11
1.0-1.1	2	53	3	14	7	27	7	18
1.1-1.2	1	54	1	15	3	29	2	20
1.2-1.3	3	60	4	18	5	34	2	22
1.3-1.4	0	60	2	20	1	35	7	29
1.4-1.5	0	60	5	24	4	38	7	35
1.5-1.6	1	61	1	24	4	42	5	41
1.6-1.7	1	63	4	28	5	46	1	42
1.7-1.8	2	67	0	28	0	46	2	44
1.8-1.9	0	67	1	28	4	50	6	50
1.9-2.0	2	70	2	30	1	50	3	53
2.0-2.5	0	70	4	33	7	57	10	63
2.5-3.0	3	75	16	46	13	68	12	75
3.0-3.5	1	77	6	51	8	76	3	78
3.5-4.0	0	77	8	58	6	81	4	82
4.0-4.5	2	81	9	65	1	82	0	82
4.5-5.0	1	82	2	67	3	85	2	84
5.0-6.0	0	82	13	77	4	88	5	89
6.0-7.0	2	85	2	79	4	92	6	95
7.0-8.0	4	93	9	86	4	96	2	97
8.0-9.0	2	96	3	89	2	97	1	98
9.0-10.0	0	96	3	91	2	99	0	98
>10.0	2	100	11	100	1	100	2	100
Total	58		123		113		125	

20152851

Arthur D Little Inc

068ETX02107

TABLE 2

SUMMARY DATA FROM A. D. LITTLE

Sample No.:	22260P-1	22260P-2	22260P-4	22260P-5
Total Fibers Counted	57	123	113	125
Arithmatic Means				
Length (μ)	2.59	4.34	2.76	2.79
Width (μ)	0.26	0.39	0.15	0.24
Average of Aspect Ratio	15.85	15.86	22.50	13.39
Mass (10^{-12}g)	0.5218	2.0464	0.1925	0.4982
Geometric Means				
Length (μ)	1.38	3.11	1.97	2.07
Std. Deviation/Avg. Length	6.6	3.5	2.4	2.0
Width (μ)	0.12	0.27	0.12	0.20
Average of Aspect Ratio	12.01	11.42	16.147	10.36
Mass (10^{-12}g)	0.0571	0.7162	0.0880	0.2584
Fibers/cm^2	52,660	295,430		
Fiber Mass/cm^2 (10^{-9} g)	27.5	606.4		

J.C.Yang 4/8/76

Fig 1. Length Distribution of Tremolite Fiber on Air Filter

(Sample No. 3) (22660P-2)

(Total Fib. Count)

(Geometric mean Length)

(Std. Deviation of the Geometric mean)

TREMOLITE FIBER BUNDLES

(Handpicked from Libby #2 Product)

CLEANING

- Washing ↓ Acetone / H_2O
- Opening ↓ Waring Blender / hi speed / 2 mins
- filtering ↓
- drying ↓ Oven / 105°C / 4 hrs

MILLING

- Spec-milling ↓ 45 sec / reruffle sample every 10 sec.
- Freeze ↓ dry ice / acetone
- Wiley milling ↓ repeat 4 times

DISPERSION ± SIZING

- disperse ↓ 1 g solid / 2.5 ℓ. dist. H_2O
- stand ↓ 20 min
- decant ↓

Solid

- redisperse ↓ 1g / 2.5 ℓ. distilled H_2O
- stand ↓ in columns 20 min or over
- decant ↓

Solid (saved for future regrinding)

Cloudy liq.

- filter ↓ Millipore, 0.45 μ

Cloudy Soln

- dilute ↓ 2X / dist. H_2O
- stand ↓ 20 min
- decant ↓

Solid

Cloudy Soln

- filter ↓ Millipore 0.45 μ

Crude Product

FIGURE 4 - TREMOLITE PREPARATION (Continued)

J.C. Yam
3/23/76

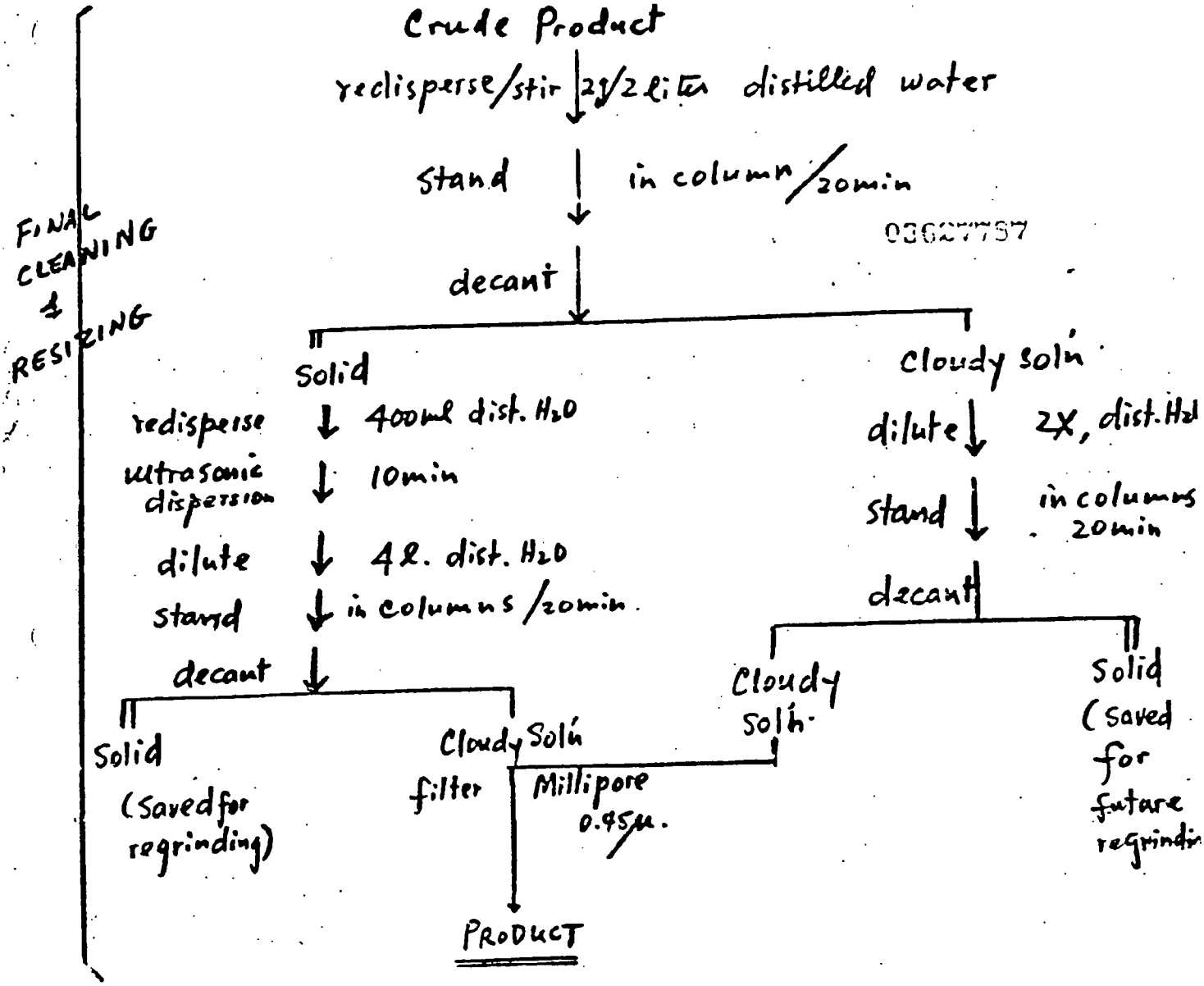
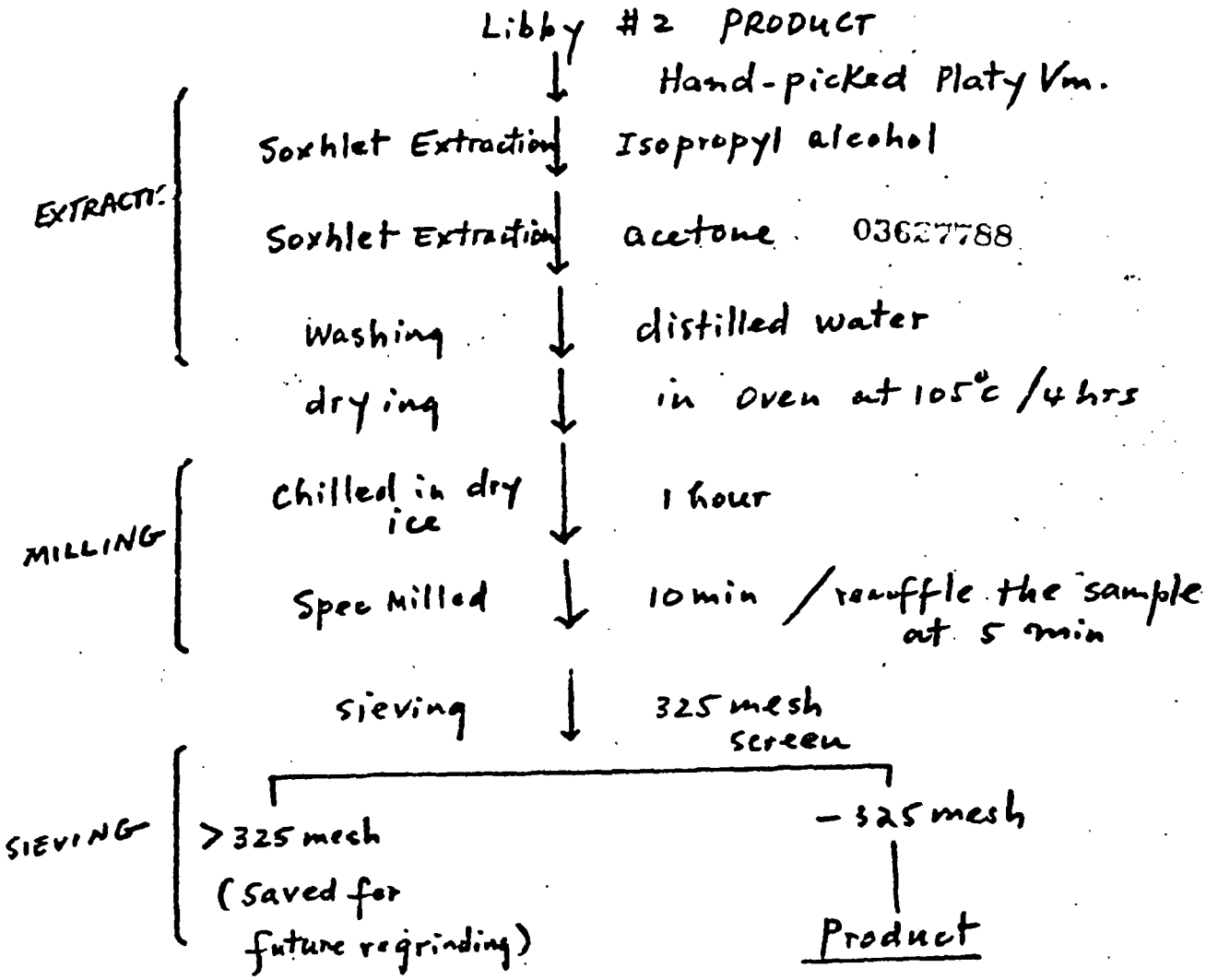


Fig. 5

VERMICULITE PREPARATION



J.C. Yang /3/24/.

Fig. 6

PROPORTIONING

03627789

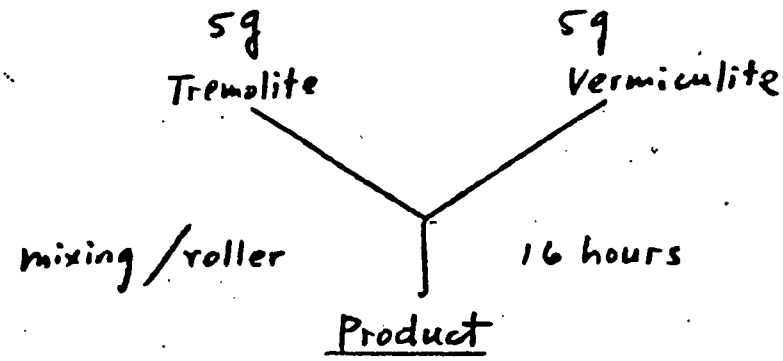


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Fig 7 Length Distribution of Tremolite Fiber Prepared (22260P-4)

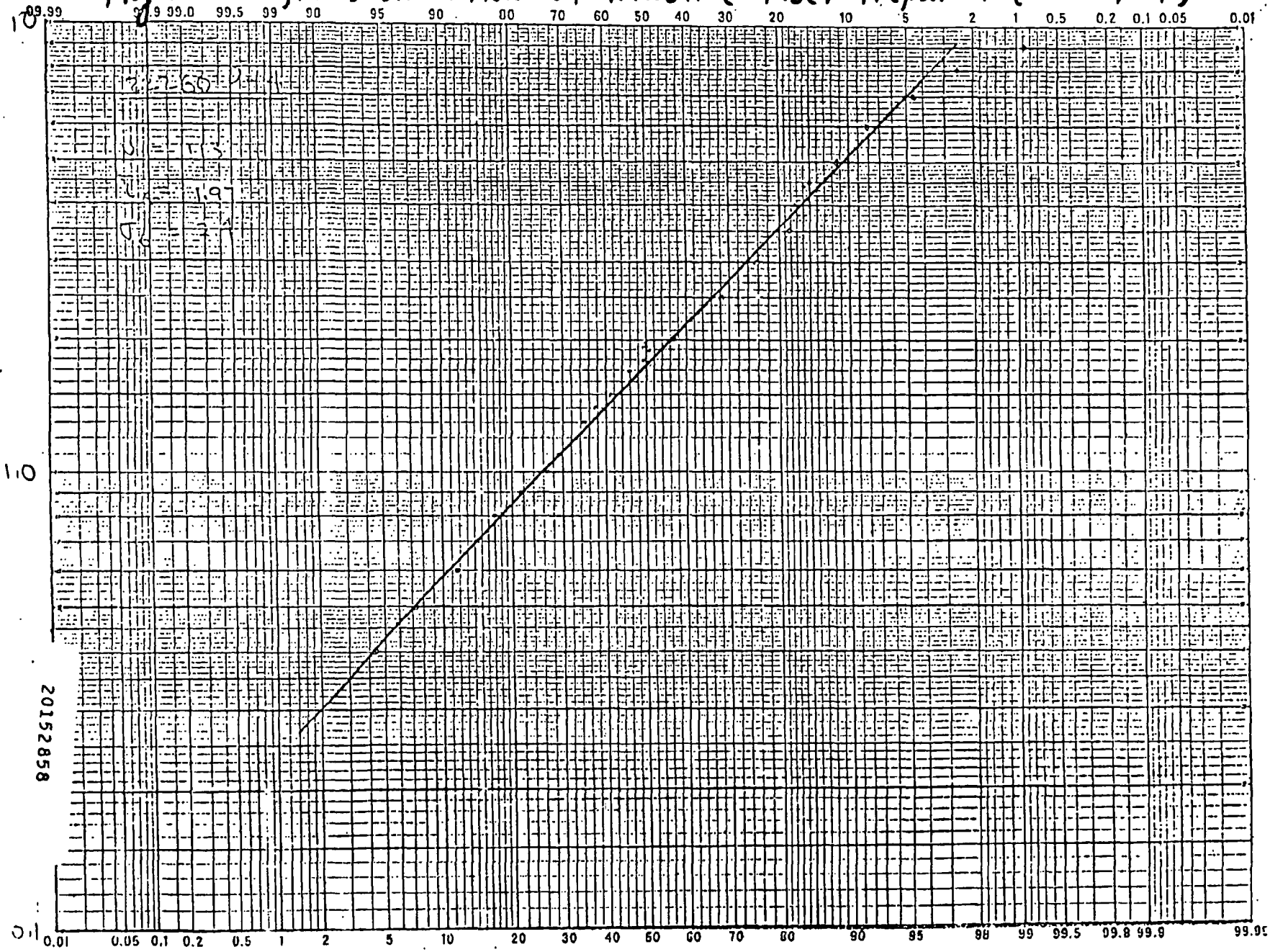

Fig 8 Length Distribution of Tremolite Fiber Prepared for Submitted To Dr. W. Smith (22260 P-5)

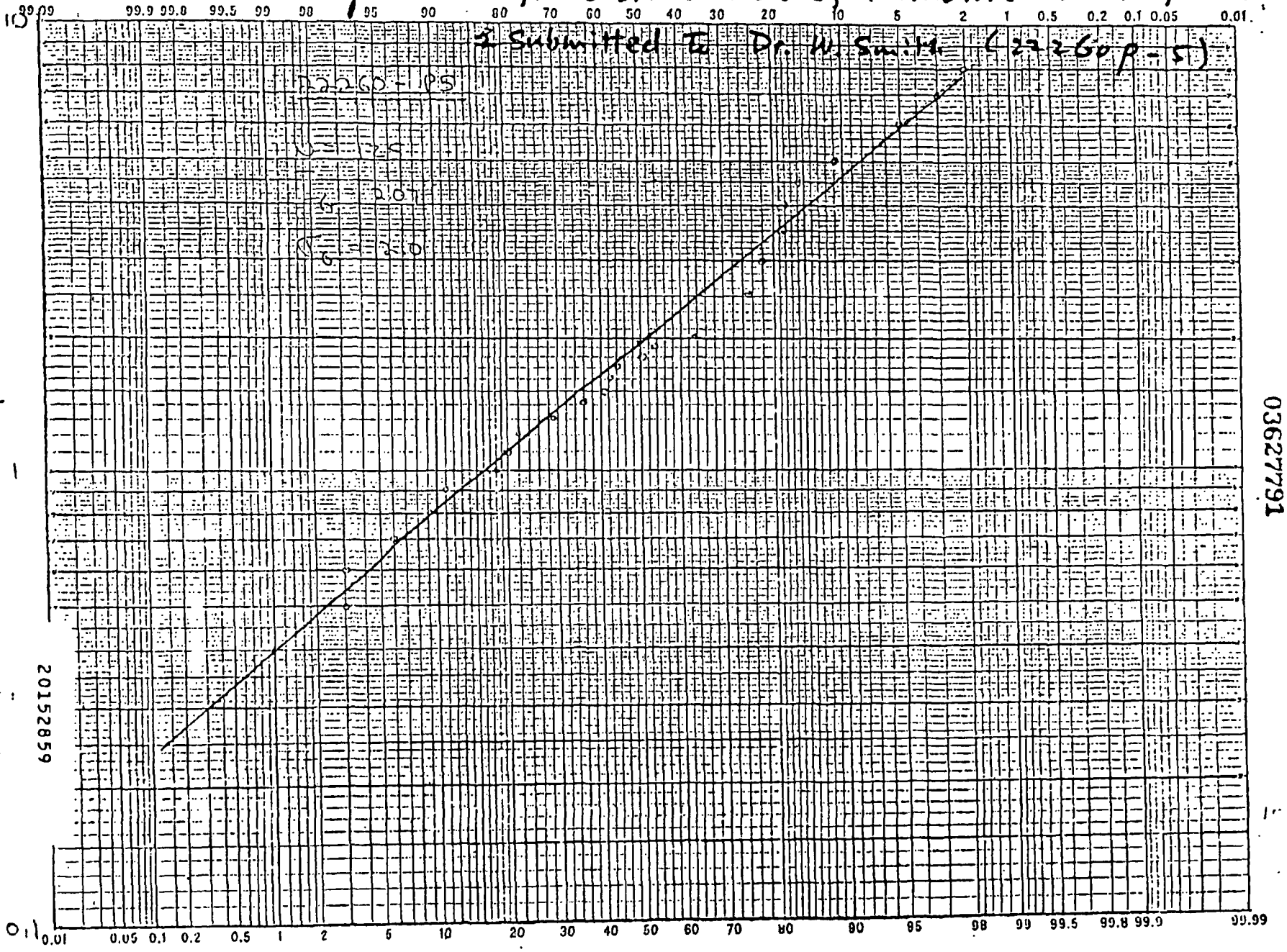

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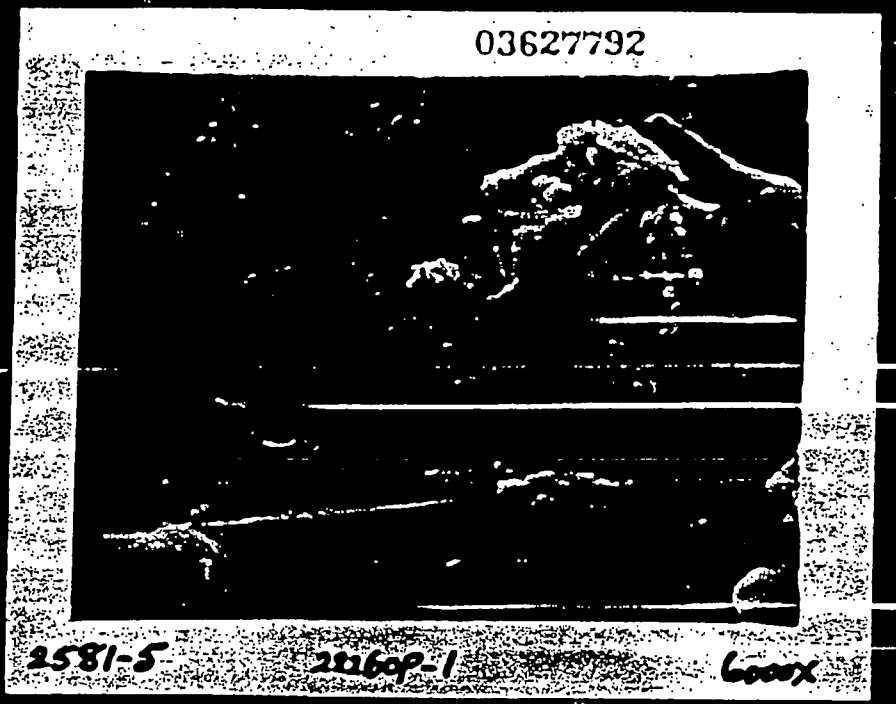
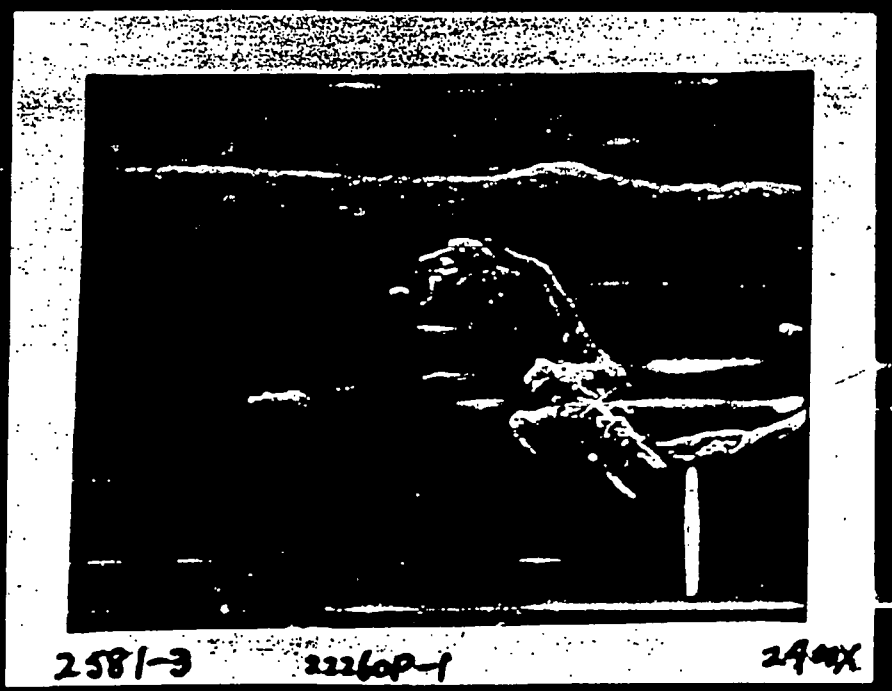
FIGURE 2
SCANNING ELECTRON MICROGRAPHS (SEM)
OF AIR FILTER #1





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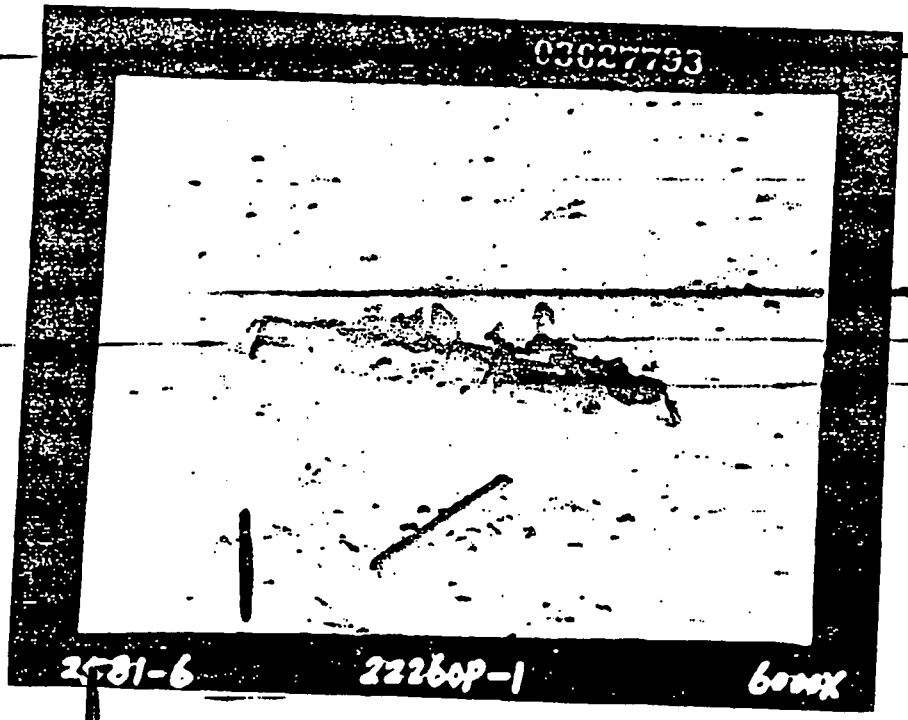

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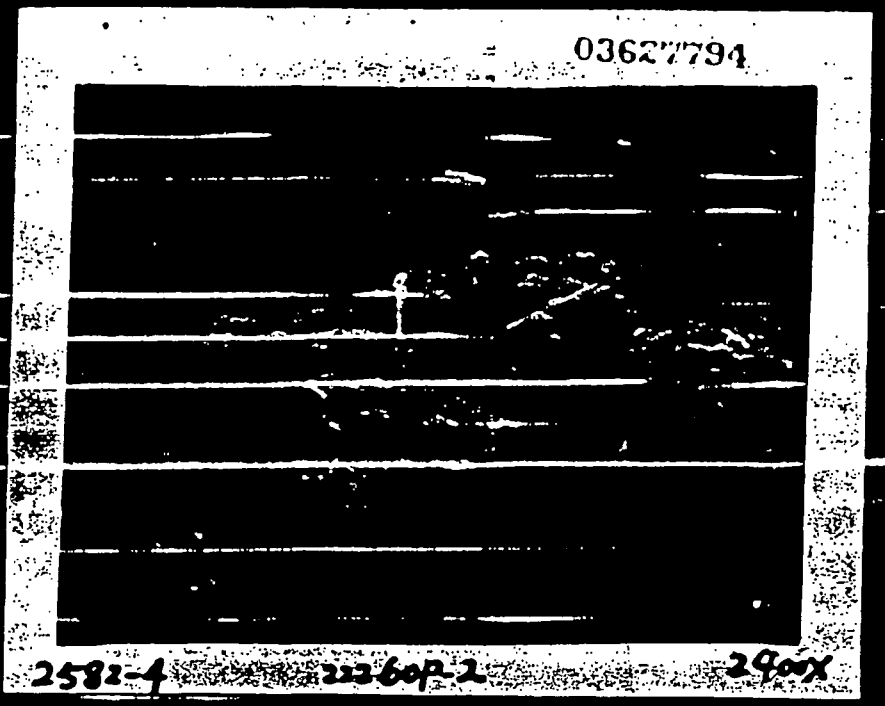
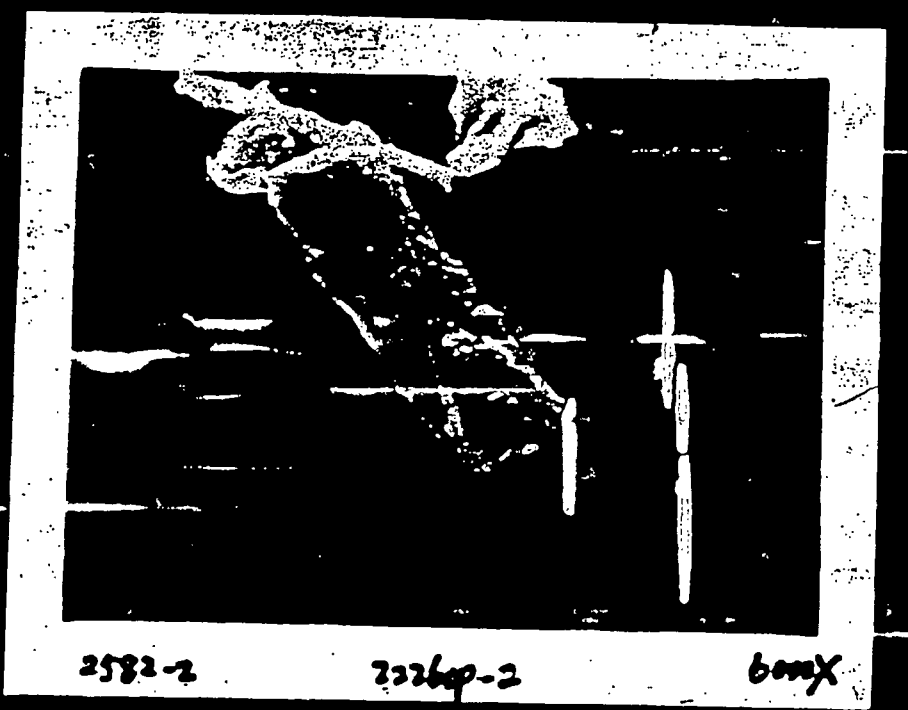
FIGURE 3
SEM OF AIR FILTER #2





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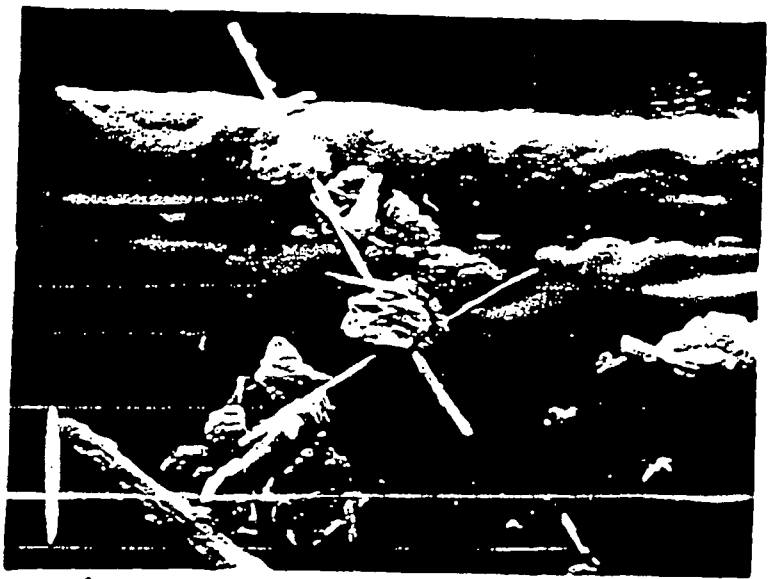
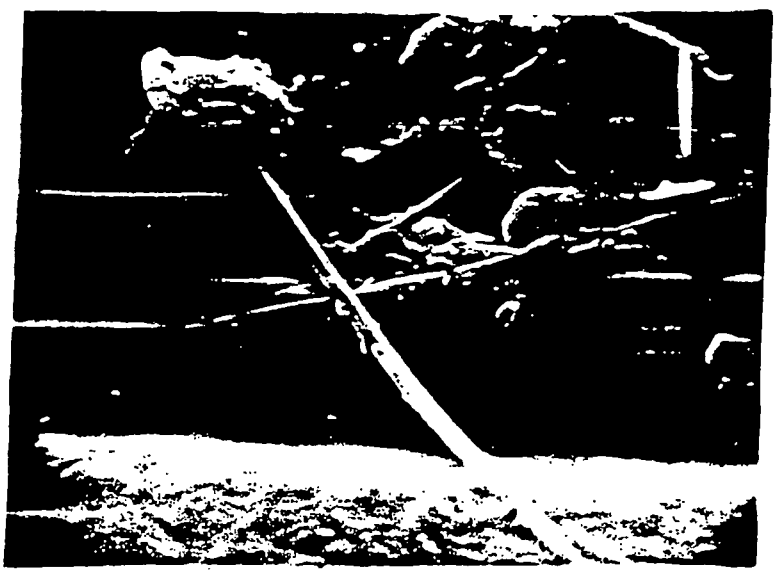


2582-6 22260P-2 2400X



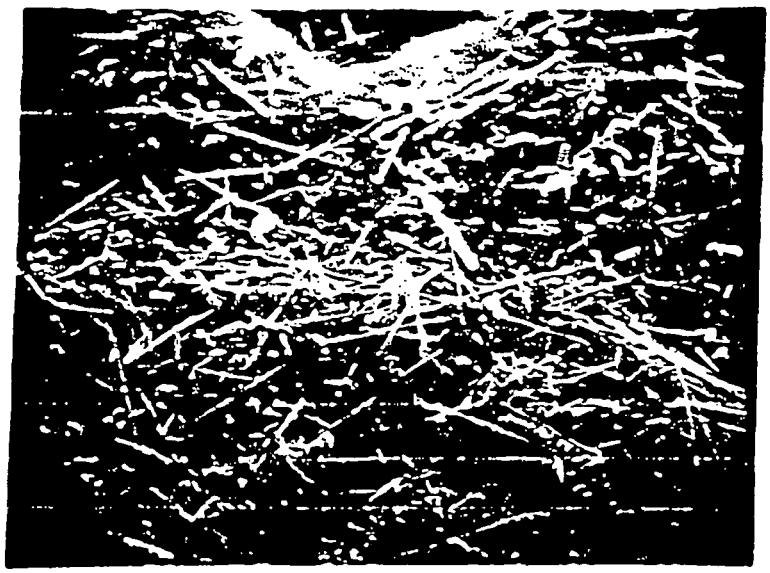
2582-2 22260P-2 6000X

20152863

FIGURE 9

SEM OF RESPIRABLE SIZE TREMOLITE FIBER PREPARED (22260P-4)

03627796



2607-5 22260P-4 2400X

2607-1 22260P-4 6000X

20152864

03627797

2607-5 222exp. 4

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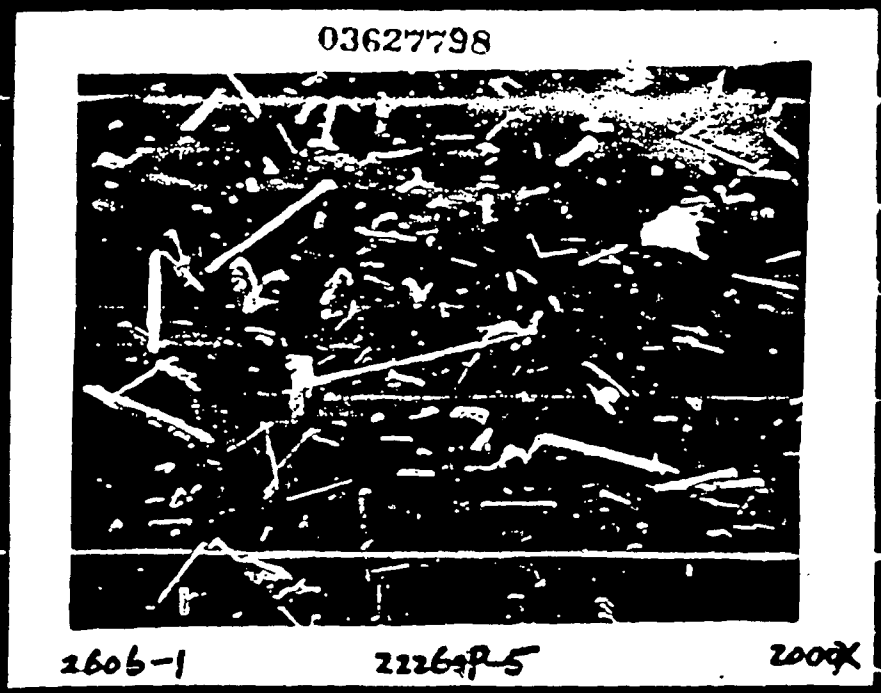
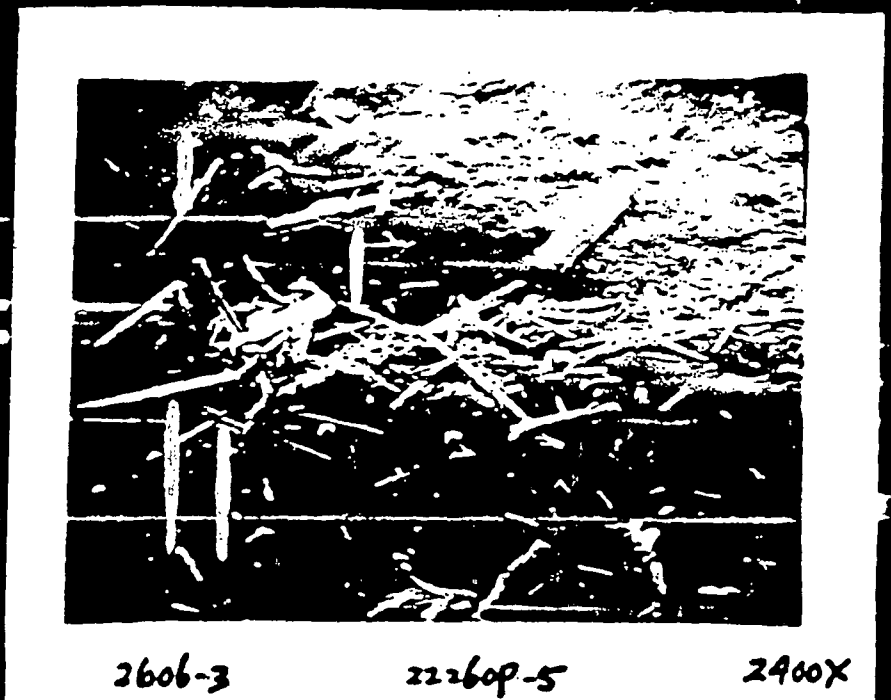
FIGURE 10

SEM OF RESPIRABLE SIZE TREMOLITE FIBER PREPARED AND SUBMITTED TO DR. W. SMITH (22260P-5)





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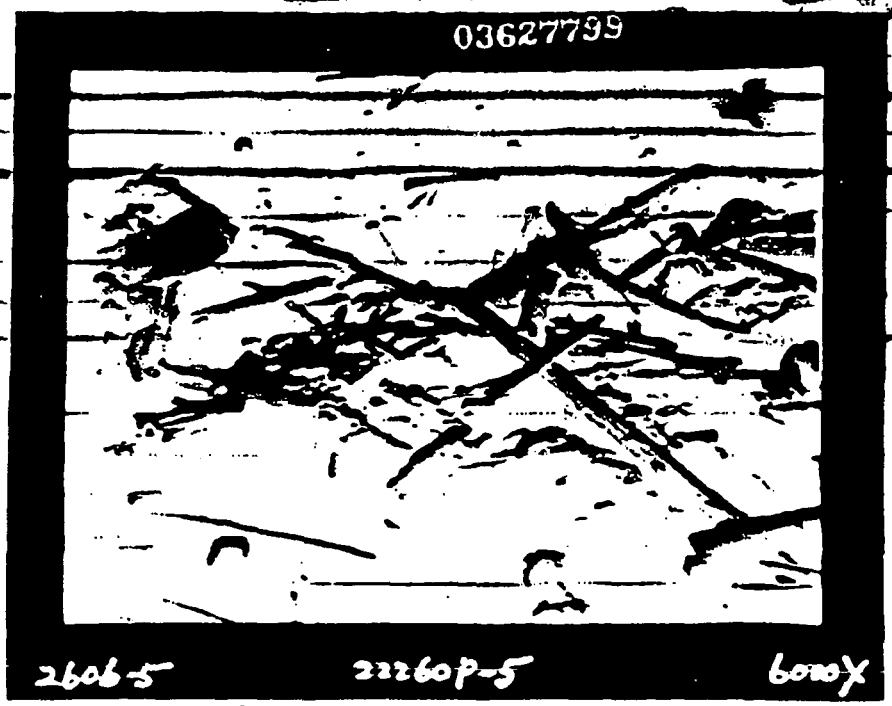

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Ref: ENF-L

February 22, 2002

BY FACSIMILE AND U.S. MAIL

Katheryn J. Coggon, Esq.
Holme Roberts & Owen LLP
1700 Lincoln Street, Suite 4100
Denver, Colorado 80203-4541
Fax: (303) 866-0200

Re: Julie Yang Deposition

Dear Ms. Coggon:

Thank you for your assistance in setting a time and place for the deposition of Dr. Julie Yang. We have agreed that the deposition will occur on August 22 and 23, 2002 in San Jose, California. I will inform you of the exact address in the near future. Pursuant to your request, we will attempt to limit the deposition to six hours per day. While EPA will make every attempt to limit the length of the deposition as discussed, we reserve the right to continue the deposition if full discovery cannot be completed. In addition, we have agreed that if Dr. Yang must postpone her deposition to go to China, W.R. Grace commits to make her available during the pendency of the discovery period.

As you are aware, Ms. Yang was the author or recipient of many documents concerning the asbestos content of Libby vermiculite. Pursuant to Paragraph 7 of the court's September 6, 2001 Order, the parties have stipulated as to the "foundation and authenticity for all written documents produced in pre-trial disclosure and during the course of discovery," unless a party objects to either with specific objections in writing within a reasonable time after receiving the document. Pursuant to this stipulation, I am assuming that W.R. Grace does not object to the

foundation or authenticity of the documents it has produced to the EPA or the United States. If this is not true, please notify me immediately, as it will obviously affect the time needed for Dr. Yang's deposition.

Sincerely,

Matthew Cohn
Legal Enforcement Program

cc: James D. Freeman, DOJ
Heidi Kukis, DOJ